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The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 67 Number 4

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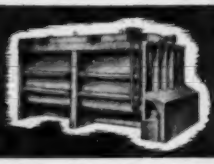

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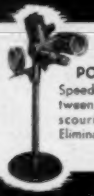

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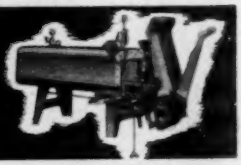

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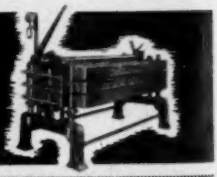

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Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1951 and pages 345–348 of the July 1950 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, 32–34 Piccadilly, Bradford, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

COMMUNICATIONS

The Combination of Wool with Acid Dyes. I—An Automatic Photometer for the Measurement of Rates of Dyeing
N. H. Chamberlain and G. H. Lister

Some Aspects of the Drying and Heating of Textiles
VI—Modifications produced by Thermal Treatments
J. M. Preston, M. V. Nimkar, and S. P. Gundavda

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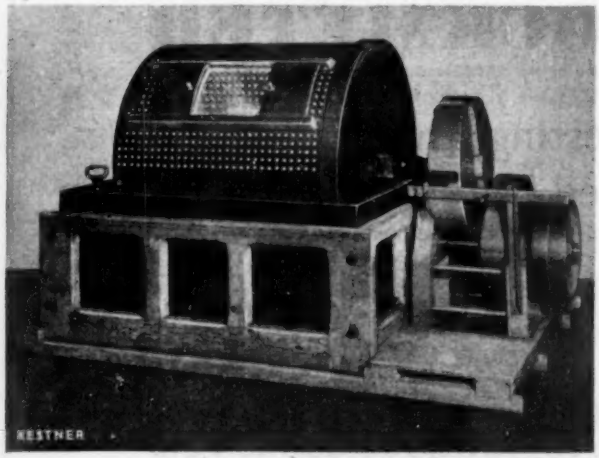

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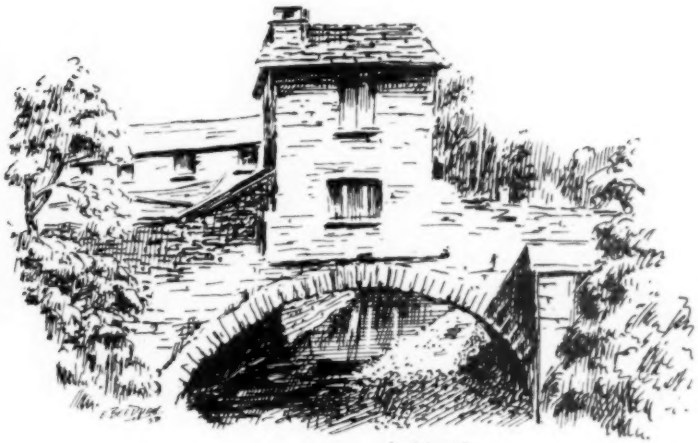

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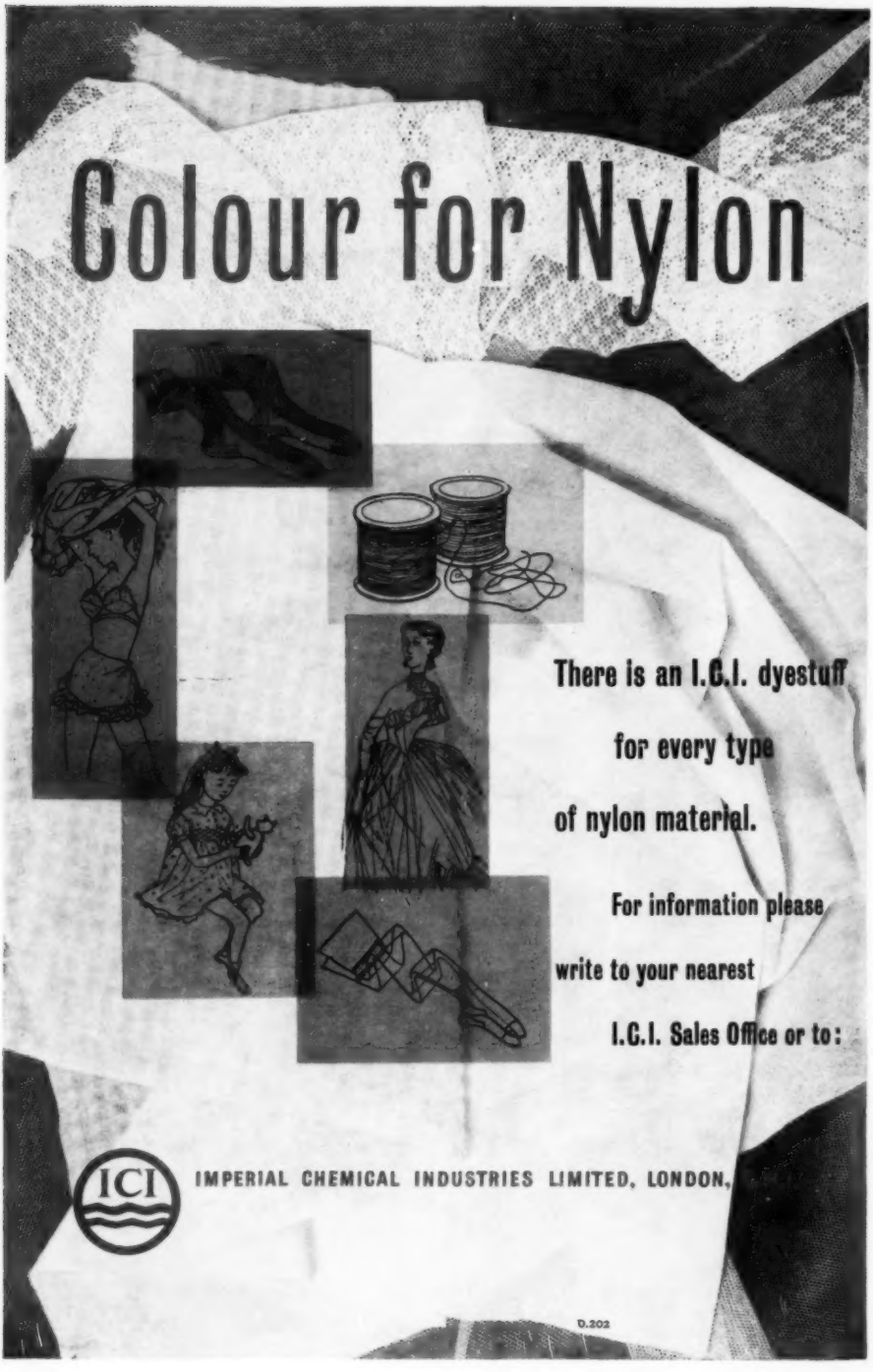
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Volume 67—Number 4 APRIL 1951 *Issued Monthly*

Proceedings of the Society

MIDLANDS AND SCOTTISH SECTIONS

Meetings held at the Victoria Station Hotel, Nottingham, on 15th February 1950, Mr. W. L. LANGTON in the chair; and at the St. Enoch Hotel, Glasgow, on 21st November 1950, Mr. R. D. ALEXANDER in the chair*

Fast Dyeings on Nylon Textiles

G. T. DOUGLAS

Nylon can be dyed with almost every range of dye, and there is no more versatile fibre as far as "colourability" is concerned. However, the dispersed acetate rayon dyes have undoubtedly been the most popular and will continue to be so. For many purposes, such as lingerie and dress goods, in view of their excellent levelling properties, compatibility in admixture, and ability to cover yarn irregularities, dispersed dyes are eminently suitable. Soluble acetate rayon dyes, e.g. Solacets, are also admirably suited for nylon dyeing. These dyes build up well, and are faster than the dispersed types to washing, in that adjacent white nylon is not stained from alkaline soaping liquors. The acid dyes provide a comprehensive range of colours of superior wet fastness to both the aforementioned ranges and also yield, in general, brighter hues. The premetallised dyes have good light fastness but do not build up well on nylon, whilst the hues are limited in their brightness. Basic dyes are bright and are readily applied, but suffer from extremely low light fastness; while direct dyes, apart from a few which almost approach acid dyes in structure, yield low colour value or only tint nylon. Not one of these ranges is fast to repeated severe washing, and to achieve this it is necessary to resort to chrome, vat, and azoic dyes.

CHROME DYES

A study of the application of chrome dyes to nylon was made, and it was soon evident that they were difficult to chrome by normal wool techniques. Increased quantities of reagents and an increased time of processing to effect complete metallisation yielded only limited success and had the added disadvantage that stripping of the dye by acid dichromate was accentuated.

Analysis of the problem suggested that the difficulties arise because—

(*a*) the absorptive capacity of nylon for dichromate ions is much less than that of wool;

(*b*) in contrast to wool, nylon is incapable of readily reducing dichromate anions to chromic cations, which are required before chelation with the dye can take place;

(*c*) chroming with chromic salts, such as chromic sulphate or chromic fluoride, is impracticable, because nylon does not readily absorb metallic cations.

Investigations have shown that, while the absorption of dichromate ions is low, it is nevertheless sufficient for complete metallisation of most dyes. It follows, therefore, that in the afterchrome method the reduction step is the principal difficulty. This has been overcome by the simple expedient of introducing a reducing treatment after the chroming stage. This not only ensures maximum metallisation of the dye within the nylon, but also completely removes residual dichromate within the nylon, which is not removed by simple washing treatments and is liable to produce serious defects in the dyed material. These are—

(1) On exposure to sunlight the residual dichromate is photo-reduced, with a corresponding degradation of the fibre.

(2) There is a possibility that dichromate may be removed in sufficient quantity by perspiration to produce skin irritation.

Not only does the special technique described in this paper completely eliminate these defects, but it also enables milder conditions of chroming to be employed, in which the amount of dichromate and the acidity of the chroming bath are reduced.

* This paper was also given at the Second Canadian Textile Seminar, held at Queen's University, Kingston, Ontario, and published by arrangement in the *Canadian Textile Journal*, 67, 47 (29th Sept. 1950).

As a class, the chrome dyes have poor redistribution properties; hence, to ensure level dyeing, the absorption of dye must be carefully controlled. For maximum penetration and levelness of result, the majority of the chrome dyes are applied as follows—

The dyebath is set with the requisite quantity of predissolved dye, 0·5–2·0% ammonia (sp. gr. 0·910), and 3·0% ammonium acetate. Dyeing is begun cold, the temperature slowly raised to 95–100°C., and dyeing continued for at least 45 min. To complete exhaustion, a gradual addition of up to 4% formic acid (85%) is made. Chroming is effected in a bath set with 4% formic acid (85%) and 0·5–2·0% potassium dichromate, depending on depth of dyeing. These quantities are based on a liquor ratio of 40 : 1. After chroming for 1 hr. at 95–100°C., dissolved sodium thiosulphate, approximately four times the quantity of dichromate used, is added to the dyebath, and 20–30 min. allowed to complete the reduction of dichromate within the nylon and produce maximum metallisation of the dye.

When heavy dyeings are being chromed, a trace of dye present in the chroming bath may cause flocculation of colloidal sulphur. In machine dyeing, especially, this precipitation of sulphur is objectionable. The precipitation can be avoided by an addition of 0·3 g. dissolved glue per litre of chroming liquor, immediately before the addition of sodium thiosulphate. Alternatively, adjusting the bath to a pH of 4·5 is also effective. Before drying, it is advantageous to give the nylon a light scour.

Only a few members of the chrome dye range are not applicable to nylon by this method.

In their dyeing behaviour on nylon, chrome dyes exhibit all the characteristics of acid dyes. The individual rates of dyeing are of little value for predicting the behaviour of dyes in admixture.

The majority of chrome dyes possess excellent building-up properties on nylon, but even at 95°C. have poor redistribution properties. As a class they have poor temperature range properties, and in general their application is not feasible below 90°C. For piece goods, closed winches or jigs should be employed; and for yarn, pressure circulating machines are ideal. Yarn irregularities are not covered by chrome dyes; in this respect the chrome dyes behave in the same way as the acid dyes. Some members of the range are also sensitive to traces of iron and copper, yielding hue inconsistencies.

Chrome dyes are very much faster to wet treatments on nylon than they are on wool. Selected dyes are capable of withstanding five separate 30-min. treatments at 95–100°C. in 5 g. soap and 3 g. soda ash per litre. A combination worthy of note for the production of mode shades is—

Solochrome Yellow C
Solochrome Red ER
Solochromate Fast Blue B

Nylon delustred with titanium dioxide is fairly rapidly degraded on exposure to sunlight. Experiments have shown that, if such nylon is treated with as little as 0·05% potassium dichromate and then reduced with sodium thiosulphate, the material is nearly four times as resistant to light as the untreated nylon. Because the recommended chroming process involves a similar treatment, chrome-dyed material is also more resistant to light than untreated delustred nylon. This protective action of chromic salts makes chrome dyes very suitable for use on materials which are liable to be subjected to prolonged daylight exposure. A range of colours excepting greens can be produced with light fastness figures of 5 up to 6–7 on the Society's scale of 1–8 (*B.S.* 1006, Part 2).

An extension to this work has shown that copper complexes of the chrome dyes yield, in general, a range of dyes of higher light fastness, although the wet fastness properties are not so high, but are still superior to those of acid dyes. Cationic copper, like cationic chromium, is not taken up readily by nylon; so the simple expedient of preparing a salt containing copper in the anion was resorted to. Copper sulphate was treated with sodium bisulphite in the presence of sodium chloride to give sodium cuprochloride $NaCuCl_2$. After dyeing with the chrome dyes, by the method described above, the nylon was treated for 30 min. at 95°C. in a bath containing 2% sodium cuprochloride. The copper anion was readily adsorbed on the end amino groups, and chelation was effected without any reduction treatment as in the case of the dichromate.

Cobalt complexes of chrome dyes yield a few interesting colours but in general these possess low light fastness. The cobalt is introduced by employing sodium cobaltinitrite as an aftertreatment as for the formation of the copper complexes. As this chemical possesses quite strong oxidising properties, certain of the chrome dyes suffer oxidative degradation. This cobalt aftertreatment is, however, of very limited interest commercially.

VAT DYES

To augment the range of fast-to-boiling chrome dyes, it is necessary to employ selected vat dyes. Application by normal methods fails to give satisfactory results, as the alkaline leuco compounds possess slow diffusion rates into nylon and in many cases only slightly tint the fibre after normal dyeing periods. Nicol[1] showed that application could be effected if high-temperature dyeing was resorted to. By using caustic soda and sodium formaldehydesulphoxylate and dyeing at 90–95°C. for 45–60 min. good colour value could be realised. Exhaustion is improved by the addition of sodium chloride. For oxidation, air, percarbonate in the soaping bath, or persulphate and acid may be employed. Blues of the indanthrone type are very liable to be partly destroyed, greening of the hue occurring, and in some cases a grey shade may be caused by the high temperature. Certain halogenated and dibenzoylamino vat dyes also suffer from degradation under alkaline reducing conditions at high temperatures.

In general, the vat dyes have low light fastness on nylon, as they are not aggregated to the crystalline state, in which they are present when dyed on cellulosic fibres. An analysis of the results

of daylight exposures shows that a definite correlation exists between the light fastness realised on nylon and the chemical constitution of the vat dyes. Table I gives the light fastness of the main constitutional dye groups.

TABLE I

Chemical Type	Number of Homogeneous Dyes tested	Light Fastness Range (on S.D.C. scale)
Thioindigoid	5	1 to 1–2
Pyranthrone	3	1 to 2
Phthaloylacridone	4	1 to 3–4
Benzoylaminoanthraquinone	6	2–3 to 3–4
Dibenzanthrone	13	3–4 to 5–6
Anthraquinone–carbazole ...	13	4–5 to 5–6
Indanthrone	7	5–6 to 6–7

A further interesting generalisation is that all dyes which are known to accelerate the tendering of cellulosic fibres on exposure to light have a very low light fastness on nylon.

The vat dyes in Table II are of especial interest on nylon as they possess good light fastness and, of course, are fast to repeated soap and soda boiling.

TABLE II

Caledon Vat Dye	Light Fastness (on S.D.C. scale)
Gold Orange 3G	5–6
Brilliant Violet 3B ...	4–5
Dark Blue G	4–5
Jade Green XN	5

A typical recipe for the application of these vat dyes to nylon is given below.

The dye is vatted in full volume at 90–95°C. for 5 min. in a solution containing 10 c.c. caustic soda (68°Tw.), 1 g. sodium hydrosulphite, and 2 g. sodium formaldehyde-sulphoxylate per litre. Dyeing is commenced at 95°C. and is continued at this temperature for 45–60 min. During this period up to 50% common salt (based on weight of material) may be added to effect exhaustion. The goods are rinsed, and then oxidised in a solution containing 2 g. sodium perborate per litre for 30 min. at 40°C., rinsed, and treated for 10 min. in 2 g. soap or Lissapol C per litre.

SOLUBILISED VAT DYES

The solubilised vat dyes suffer from the same defects in light fastness as their parents. They are difficult to apply, also requiring high temperatures to effect penetration of the nylon. The vat esters are best applied from an acid Formosul dyebath, and the parent vat dye is regenerated by treatment in a solution containing 0·25% sodium nitrite and 0·25% sulphuric acid (168°Tw.) at 50°C. for 15 min. A patented process[2] recommends a steaming after-treatment; this, causing some aggregation, does effect an improvement in light fastness, but in all cases the light fastness of the vat esters is inferior to that obtained on the cellulosic fibres. Because of application difficulties, the leuco vat esters are not likely to find a very great outlet on all-nylon fabrics.

AZOIC DYES

The view generally held is that azoic combinations cannot be applied to nylon by the normal techniques employed for their application to cellulosic fibres, for the following reasons—

(1) The naphthol body has poor affinity from alkaline solutions at low temperatures

(2) The diazonium chloride does not penetrate into the naphthol-treated nylon.

A reinvestigation of the application of azoic dyes to nylon yielded some interesting results—

(*a*) UPTAKE OF INERT COUPLING COMPONENT—The Brenthol range was applied to nylon both as free hydroxy compounds and as the sodium salts, and the absorption measured at temperatures of 20–100°C. Over a period of 45 min. maximum absorption was realised at 80–85°C. The absorption of the hydroxy compounds at 85°C. was generally appreciably lower than that of the napthoxide ion when sodium chloride, which increases exhaustion, was added to the dyebath. The uptake of Brenthols on nylon was found to be as high as on viscose rayon when employing the optimum temperatures of application for each fibre. Table III gives a selection to illustrate the absorption of Brenthols on nylon (2 g. Brenthol per litre at 85°C. and a liquor ratio of 20 : 1 for 45 min.).

TABLE III

Brenthol	Exhaustion (%)
AS	39
BA	51
BT	87
CT	76
FR	40

(*b*) COUPLING—From a consideration of the chemical structure of nylon it was to be expected that the diazonium chlorides should be totally incapable of penetrating nylon from acid solution, and in practice this was found to be the case. Treatment of Brenthol-dyed nylon yielded little or no coupling with a diazotised base even at slightly elevated temperatures. Any coupling which took place was mostly on the surface of the fibre, and the tint produced was fast neither to soaping nor to rubbing. The free base was, however, readily absorbed from an alkaline dyebath even at low temperatures. Diazotisation of the base *in situ*, by treatment in sodium nitrite and hydrochloric acid for 30 min. at 25°C., produced coupling. Maximum coupling was complete within this period. Increase in the temperature of diazotisation yielded increased rate of coupling, but duller hues were obtained. At this stage fast-to-rubbing results were obtained with all azoic combinations.

(*c*) SOAPING—Soaping is necessary to remove excess base or uncoupled Brenthol, but it was noticed that with many combinations a bright orange product was bleeding off during the scouring treatment. Investigation showed that this was nitrosolated Brenthol. Brenthols of the 2:3-hydroxynaphthoic acid series yielded bright orange, and the carbazole types green or violet, nitroso bodies which were not fast to light.

(*d*) ELIMINATION OF NITROSOLATED BRENTHOL—Alkaline scouring agents were tried, but failed to effect complete removal of the nitroso-Brenthol. Decreasing the sodium nitrite content of the diazotisation bath diminished the amount of nitroso

compounds, but also reduced coupling. The addition of formaldehyde to the Brenthol bath increased absorption of the Brenthol and was completely effective in eliminating the formation of nitroso bodies, but other defects were apparent. There was a considerable loss in depth of dyeing obtained, the rubbing fastness deteriorated, many of the azoic combinations bled on soaping, and there was a considerable amount of uncoupled Brenthol–formaldehyde reaction product which was incapable of being removed by a soaping treatment. For these reasons formaldehyde was not considered to be really beneficial. The most effective method evolved was to employ twice the theoretical quantity of base to Brenthol calculated on the absorption figures of the components on the nylon. By this expedient, the formation of nitroso-Brenthol was kept to a minimum.

Two further points may be emphasised here. On giving the azoic-dyed nylon a normal soaping aftertreatment it was noticed that—

(1) Colour changes which occur with certain azoic combinations on cellulosic fibres were completely absent in nylon. This is due to the fact that no aggregation of the pigment particles takes place in the nylon, and aggregation cannot be effected even by pressure steaming or by any swelling treatment. However, when certain nitro bases were used, reduction appeared to occur to give marked colour changes.

(2) The rubbing fastness decreased. This is due to the pigment being forced to the surface; only when it does reach the surface is aggregation possible, yielding a reduced fastness to rubbing. Azoic combinations of high molecular weight are less prone to migrate, and with these good rubbing fastness is obtained. Also the more complex the molecule the greater the washing fastness.

In outline, the modified method consists of simultaneous high-temperature absorption of dispersed base and Brenthol from a caustic alkaline solution, followed by cold diazotisation and coupling *in situ*. Surface pigment, uncoupled intermediates, and deleterious by-products, such as nitroso-Brenthols, the formation of which, by increasing the ratio of base to Brenthol, is kept to a minimum, are removed by a thorough soaping treatment. In detail, azoic dyes are applied to nylon as follows—

The Brenthol (1 g.) is pasted with 4·5 c.c. Azomel A (50%) and 4·5 c.c. hot water. To the smooth paste 30 c.c. hot water is added, and the dispersion brought to the boil, when an addition of 4·5 c.c. caustic soda (68°Tw.) is made and heating is continued till solution is complete.

The base (1 g.) is pasted with 1 c.c. Dispersol VL, and then 50 c.c. hot water is added gradually with constant stirring.

The dyebath is prepared so as to contain 20 g. common salt per litre and sufficient caustic soda (68°Tw.) to produce a final concentration of 5 c.c. per litre. With Brenthol AN and Brentamine Fast Blue B Base, 2 g. Brenthol and 4 g. Base per litre are employed to yield a full navy blue when dyeing from a 20 : 1 liquor. Dyeing is commenced at 85°c. and continued at this temperature for 45 min. The nylon is rinsed, and then treated in a cold solution containing 15 c.c. hydrochloric acid (32°Tw.) and 6 g. sodium nitrite per litre for 30 min. to effect diazotisation and coupling. Finally, the goods are treated in 2 g. soap per litre at 95°c. for 30 min.

In all cases the dyeings are characterised by excellent colour yield, but the brightness does not equal that realised on cellulosic fibres. Selected combinations are fast to boiling soap and soda as far as loss in depth and change of hue are concerned. Adjacent undyed cellulosic fibres are very slightly stained, but white nylon is heavily stained, more so if vigorous boiling is carried out, as azoic pigment in a very fine state of dispersion is readily absorbed by nylon. In full depths, with selected combinations, the fastness to light varies from good to excellent.

Although most azoic combinations can be applied by this method, those which yield dyeings of good wet, light, and rubbing fastness are very limited. It has been found that only those azoic combinations of the highest molecular weight, e.g. Brenthol LTR and AN and Brentamine Fast Scarlet G, Scarlet GG, Scarlet RC, Red KB, Red LTR, Red RC, Garnet GBC, and Blue B Base, yield dyeings fast to rubbing and wet treatments.

With careful selection from the chrome, vat, and azoic ranges, fast-to-boiling dyeings with good light fastness can be obtained, and in staple nylon yarns and fabrics, where the best possible fastness is called for, these fast ranges will serve the dyer well.

NYLON–WOOL UNIONS

By far the most important union in which nylon is now finding outlet is wool–nylon. Here, fast dyeings are called for, mainly for men's half hose, but fastness to boiling soap is neither required nor expected. Incorporation of nylon into wool gives a material which possesses high resistance to abrasion and also added strength over that obtainable with wool alone.

The dyeing of wool–nylon is complicated, as it is not possible to deduce any broad generalisation in the behaviour of any class of dyes. Each dye has its own characteristic behaviour, and choice must be based on the nature of the union, the fastness requirements, and the individual properties of the appropriate dyes. For fast dyeings, milling acid, premetallised, and chrome dyes provide the most suitable products for the dyeing of wool–nylon union materials.

The distribution of the dye between the two components of the union is dependent upon the depth being dyed. Dyes possess a greater intrinsic anionic affinity for nylon than they do for wool, so that when pale shades are dyed and saturation of the nylon does not occur, the nylon is dyed heavier than the wool. As the depth is increased, a concentration of dye is reached where the nylon and the wool are dyed to an equal depth. This corresponds to the saturation value of the nylon. Beyond this point, build-up occurs only on the wool component. The percentage depth at which equality is attained on the wool and nylon varies widely from dye to dye, and is dependent upon the

anionic affinity, basicity, diffusibility, and commercial purity of the dye. It is convenient to classify dyes arbitrarily on a basis of partition between the wool and the nylon at a medium depth. This classification immediately gives the dyer an indication as to which dyes will give solid dyeings in pale, medium, and heavy depths. Chrome dyes are best suited for the production of fast heavy dyeings on wool–nylon unions, as many of the chrome dyes yield equal or heavier depths on the nylon when dyed at a 3% depth. The afterchrome process must be employed, and again it is recommended that a sodium thiosulphate treatment be given to avoid all risk of dermatitis due to the presence of dichromate in the nylon. Blacks, browns, navy blues, and maroons, popular in men's half-hose, are readily obtained with chrome dyes. For pale and medium depths, the premetallised dyes are eminently suitable, but the low saturation value of these on nylon excludes their use in heavy depths. Where brighter hues are desired, it is necessary to employ the milling acid dyes, and these also are suited to the production of pale and medium depths provided a neutral dyeing technique with the aid of Carbolan Salt A is employed. However, in certain cases it is necessary to use dispersed acetate rayon dyes to augment the depth realised on the nylon to obtain solidity of dyeing on the union. This expedient tends to lower the wet fastness properties of the dyeing, but provided an after-soaping is given, a reasonable degree of fastness is realised if heavy depths are not being dyed.

Wherever wool is met, the present-day tendency is to chlorinate to achieve non-shrink properties, and the sales value of a "shrink-resistant" label is not to be neglected. All the recorded tests on 100% nylon indicate that such a treatment, particularly on subsequent exposure to sunlight, would result in very severe tendering of the nylon. A typical wet chlorination process was selected for study, a normal antichlor treatment being given after chlorination. The results are summarised in Table IV, from which it appears that preferential absorption of chlorine by the wool is causing protection of the nylon, and that chlorination can be regarded as more or less satisfactory for wool–nylon unions provided there is more than 50% wool present in the union. Selection of dyes is, of course, necessary to withstand the chlorination process, and it is to be noted that the fastness to chlorination of a dye on nylon is not necessarily the same as its fastness on wool.

TABLE IV

Chlorinated Nylon (%)	Material Wool (%)	Loss in Strength of Nylon after Light Exposure (%)
100	(unchlorinated)	37
100	—	66
50	50	44
5	95	36

NYLON–CELLULOSIC UNIONS

For the production of fast dyeings on nylon–cellulosic unions, the choice of suitable dyes for the cellulosic portion is confined to vat, solubilised vat, or azoic dyes. If the percentage of nylon is low and there is an intimate fibre blend, vats or vat esters, applied by a normal process but using higher temperatures, will give reasonable solidity. The overall light fastness will be only a little inferior to that experienced on an all-cellulosic material. If the nylon component is visually evident, the cellulosic component may be dyed with vat, vat ester, or azoic dyes at low temperatures, and the nylon cross-dyed with selected acid, premetallised, or chrome dyes. By this means solid or contrasting dyeings can be produced, the fastness being limited by the dyes applied to the nylon.

NYLON–SILK UNIONS

Silk–nylon unions are likely to be met only in limited quantities, and the fastest dyeings would be realised with chrome dyes employing a similar technique to that for wool–nylon unions. Milling acid dyes and premetallised acid dyes, bearing in mind the partition between the two fibres, will for most purposes be satisfactory from a fastness viewpoint.

Incidentally, many chrome dyes on nylon will withstand the silk degumming process and can be employed for effect threads in silk–nylon unions.

This is the position as regards the production of fast dyeings on nylon textiles to-day. The call for high fastness on nylon textiles is, as yet, not great, but the more progressive dyers are tackling these problems when the demands are made. All the processes described, except those in which limited interest has been expressed, have been used in bulk working in Britain, and certain lines, where the demand is high, are being run daily. However, in five years' time perhaps the colourist's views and recommendations may have changed completely, and a quite different story will be told.

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DYESTUFFS DIVISION
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MANCHESTER 9

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COMMUNICATIONS

Comparative Tests on the Shrink-resisting of Men's Half-hose

J. R. STEWART

The relative effects of four methods of shrink-resisting men's half-hose were evaluated in relation to the physical and chemical changes brought about and in relation to shrink-resistance and serviceability in wear. In each case, treated material was compared with untreated material of the same specification. Considerable differences between the effects of some of the four methods were found, and, in view of the difficulties encountered in works control of some of the processes, simplicity of application appears to be one of the major considerations desirable in works practice.

INTRODUCTION

In order to control as many variables as possible in this investigation, the crossbred yarn for this purpose was spun specially and the tests were carried out partly on the yarn itself and partly on conventional men's socks knitted from it.

Where the shrink-resisting process demanded it, yarn and socks (in the undyed state) were scoured before processing, but wherever possible unscoured material was shrink-resisted and then scoured, and the scouring could in some cases be of a very mild nature. One of the processes gave rise to a yellowish tan colour of the wool, which required discharging by a conventional peroxide bleach prior to dyeing.

After shrink-resisting, yarn and socks were dyed separately, but under similar conditions. Pre-metallised dyes were used in order to obtain adequate fastness on the shrink-resisted material. The yarn was tested before and after treatment for physical and chemical modification; the socks were worn under controlled test conditions by a panel of nine members, and washed centrally at specified intervals, dimensional changes being ascertained after each washing test.

PROCEDURE

The yarn spun for these experiments was made from 58s Australian crossbred lambs. A twofold yarn was made of 2/14s worsted count with 8 t.p.in. "Z" spinning twist and 6 t.p.in. "S" folding twist. The wool contained 6% of a pre-emulsified type A wool oil.

To study the effect of the shrinkproofing treatments on nylon, heels and toes of the socks were made with a reinforcement yarn consisting of single 18s staple nylon yarn spun on the worsted system from 3-denier 4-in. staple delustred nylon.

The crossbred yarn was divided into two lots, one being wound into 2-oz. skeins for treatment in yarn form; the second lot was knitted into socks of the following specification—

COURSES PER INCH	
1 × 1 ...	12
3 × 1 ...	11
Plain ...	10
WALES PER INCH	
1 × 1 ...	7
3 × 1 ...	7
Plain ...	8

KNITTING METHOD

2 ends of yarn	
4 in. ...	1 × 1 top 40 + welt courses
7½ in. ...	6 × 3 leg 80 courses
8 in. ...	6 × 3 instep and plain foot 80 courses

MACHINES USED

Harley-Kay 72 × 4½

REINFORCEMENT

Heel and toe by splicing

These two types of material were scoured, except as stated in the introduction, at 20 vol. with 2% neutral flake soap, 1% soda ash, and 0·5% tetrasodium pyrophosphate at pH 9 and 40°C. for 20 min. with occasional squeezing in a pair of rubber nip rollers. After scouring, the material was rinsed free of soap and alkali with lukewarm soft water until the wool reached pH 7.

The shrink-resisting processes used in this investigation were selected partly from prominent methods used in the industry, two English, one Australian, and one American. The process involving the use of alkali as the shrink-resisting ingredient has no industrial application in Canada as yet, but was chosen to provide an alternative to the redox principle common to the other three.

Skeins and socks were treated together in stainless steel vessels. To minimise personal bias of test personnel the processes were given code names, so that after treatment any subjective testing, such as for colour, softness, etc., should be free from influence of the knowledge of what type of process was employed.

For convenient comparison, most of the processes used are given here based on 100 g. of textile material, but in actual testing 200–300 g. was used.

PROCESS B—Unscoured material (100 g.) was treated without previous wetting-out in 3 litres of water at 5°C. containing 22 g. of commercial sodium hypochlorite of 9% available chlorine content, and sufficient formic acid and sodium formate to give a well buffered solution of pH 5. Igepal CA extra (an aryl–ethylene oxide condensation product) (10 g.), which is substantially unaffected in its wetting-out properties by the test solution, was added, and the wool goods were entered and gently worked below the liquor surface for a total of 6 min. During this time the pH was kept just below 4 by small additions of formic acid. The available chlorine in solution became exhausted after 4 min. At the end of 6 min. sodium bisulphite (10 g.) was added, and the wool treated for a further 10 min., rinsed cold, neutralised in a weak bicarbonate bath, and, after rinsing, scoured.

The pH of the baths was controlled by means of a Coleman glass-electrode electrometer. The chlorine content was titrated with 0·1 N. sodium arsenite in presence of bicarbonate anion, using starch–iodide as external and internal indicator.

PROCESS C—Unscoured wool (100 g.) was treated without previous wetting-out in 2·5 litres of water at 20°C. containing—

2 g.	...	Potassium permanganate
42 g.	...	Sodium hypochlorite (9%)
7 g.	...	Calcium chloride (anhydrous)
3 g.	...	Zinc sulphate (crystals)
0·5 g.	...	Igepal CA extra (General Dyestuffs Corpn.)

Treatment was continued for 22 min. After 20 min. all available chlorine had reacted. As in Process B, a slight excess of sodium bisulphite was added, and the pH, which was kept at 6 during the treatment with chlorine and permanganate, was adjusted to 4. The treatment with bisulphite continued until the brown colour of the wool due to deposited manganese dioxide was discharged. The wool was then rinsed thoroughly, neutralised with bicarbonate as above, and scoured.

In both processes (B and C) the scouring required was very slight, and consisted of 0·5% ethylene oxide condensate and 0·5% ammonia (sp. gr. 0·900) for 10 min. at 40°C. followed by two lukewarm rinses.

PROCESS M—Scoured wool (100 g.) was treated for 15 min. in 2 litres of water at 20°C. containing 2 g. of Ional ST (Clough) (a non-ionogenic surface-active agent) and 7 ml. of hydrochloric acid (20° Bé.).

Then 15 g. of sodium sulphamate was added and the goods were worked a further 10 min. at 20°C. After this time 4 g. of "high test" calcium hypochlorite (70% available chlorine) previously dispersed in a little water was added, and the pH kept just below 4 during 15 min., whilst the goods were worked below the liquor surface. At the end of this period, titration showed exhaustion of the available chlorine in solution.

The wool was next treated in a fresh bath containing a 1% solution of 100-vol. hydrogen peroxide and 0·5% ammonia (26%). The goods were worked for 10 min. at 50°C., and then two lukewarm rinses were given. The wool then exhibited a yellowish tan colour, which was discharged by bleaching with 3-vol. hydrogen peroxide, 0·5% ammonia, and 0·5% Stabiliser C, a mixture of sodium oxalate and sodium pyrophosphate. The bleach was carried out for 3 hr. at 45°C., and followed by two final rinses. The bleach will not be necessary in practice except for pale blue and green shades and bright dyeings.

PROCESS R—Scoured wool (160 g.) was treated in a solution containing—

30·5 g.	...	Caustic potash in
188 ml.	...	Water run into
2800 ml.	...	Methylated spirits (85% ethyl, 15% methyl alcohol) containing
40 g.	...	Glycerol

The wool was worked in this alcoholic solution for exactly 2 min., immediately squeezed off through rubber nip rollers, entered into a bath containing—

45 g.	...	Sulphuric acid (95%) in
2800 ml.	...	Methylated spirits

and worked for 10 min.

The goods were then rinsed in a fresh bath of running soft water (cold), neutralised in a weak bicarbonate solution, and rinsed again.

EVALUATION

The materials—yarn and socks—treated by the four processes were dried and compared against scoured, scoured and bleached but untreated, standards of the same origin. The effect of the treatments on colour, lustre, softness, and "loftiness" is summarised in Table I (changes were similar for yarn and half-hose).

TABLE I

Process	Colour	Lustre	Softness	Loftiness
B	Slightly yellower	No change	Softer	Loftier
C	Slightly yellower	No change	Much softer	Loftier
M	Tan*	No change	No change	No change
R	Yellower	Very lustrous	Harsh and "scroopy"	Less lofty

* Refers to treated wool before bleaching with 3-vol. peroxide.

Next, the extent of physical and chemical modification, and possibly damage, to the treated wool was ascertained, using the scoured, untreated materials as standards of comparison.

The chemical modification tests employed were—

(1) Microscopic scale examination, fibres mounted dry and in 50% aqueous glycerol
(2) Allwörden reaction
(3) Pauly diazo reaction
(4) Kiton Red G stain.

Standard test procedure was used. The results are shown in Table II.

TABLE II

Process	Scales	Allwörden ("Elasticum")	Pauly Test (Damage)	Kiton Red (Damage)
B	No damage	Absent	Isolated	Isolated
C	Slight blunting	Absent	> B	> B
M	No damage	Absent	< B	< B
R	Blunting	Absent	> C	> C

It will be seen that the scale appearance, Pauly test, and Kiton Red test corroborate each other, whilst the Allwörden reaction gives the result typical of "chlorinated" wool even in the case of the alkali-processed samples. On the whole, least chemical modification seemed to have been sustained by wool processed in B and M, whilst R shows distinct modification and change in physical properties.

It was now desirable to assess the effect on mechanical strength and extensibility of the treated samples. With unscoured and scoured yarns as comparison, tensile strength and total extension at break were ascertained in each case. The conditioned yarn was tested by a constant rate of loading (inclined-plane) single-thread tester, Model IP–2 (Suter), taking the arithmetical mean of 40 readings in each case. It is to be expected that, if any treatment brings about a change in count of the yarn owing to shrinkage, swelling, stretching, or attack on the fibres, the strength will vary.

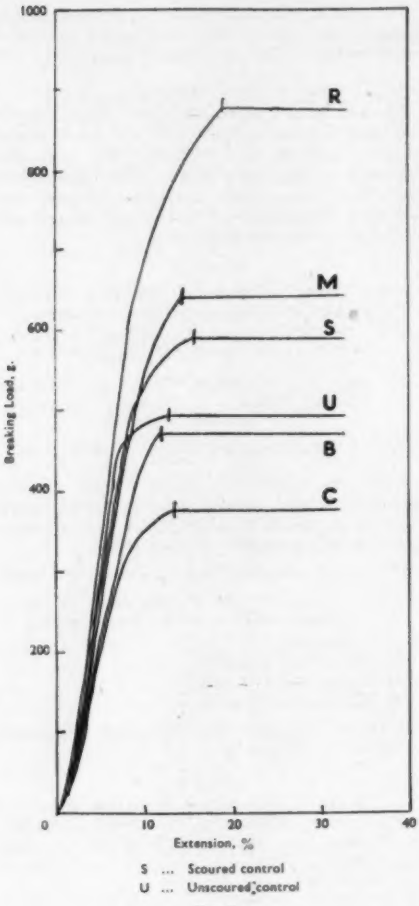

FIG. 1

Therefore the count variation was determined in each case, and the necessary corrections are shown separately (Table III). For easier reference, the results are also shown graphically (Fig. 1).

TABLE III

Material	Count (B.W.)	Break Load (g.) Measured Value	Break Load (g.) Increase (corr. for count)	Extension (%) Measured Value	Extension (%) Increase (corr. for count)
Grey yarn ...	6·96	489	—	12	—
Scoured yarn ...	6·85	591	+ 3	14·6	+ 2·4
B and scoured	7·26	470	+ 2	15·3	+ 3·8
C and scoured	7·26	376	− 92	12·4	+ 1·1
M and bleached	6·22	640	+ 90	14·8	+ 3·4
R and scoured	5·95	870	+ 300	18	+ 4·0

The corrections for break load are based on the count of the grey yarn, so that e.g. "+ 3" in the case of the scoured yarn refers to the fact that a yarn of the count 6·85 would be expected to show 588 g. break load, while the actual load found is 591, three grams higher. Negative values indicate that the yarn in question showed less than proportionate strength. The corrections for extension are similarly computed but expressed on the original percentage scale.

The scoured yarn shows the increased strength to be expected from relaxation and removal of fibre lubricant. Two of the processes (B and C) increase the yarn count probably owing more to loss of some of the springiness of the yarn than to actual fibre attack (see Table II). The two other processes (M and R) yield a thicker yarn after processing. In the case of process R the resulting increased strength (300 g. corrected for count) is out of all proportion to the increase in yarn thickness, and it must be assumed that the increased harshness and stiffness of the fibres were responsible for this. The high extension at break would also point to the yarn holding together despite some fibre slippage (cf. shape of R graph on Fig. 1).

It is quite clear that Process C is worst in its effects on strength and extension. This process also yields the softest fibre, whilst the physical characteristics of the Process R yarn with respect to stiffness, lustre, and scroopy handle approximate to those of mohair or "lustre" yarns.

DYEING

Pairs consisting of one treated and one untreated sock were dyed in the same bath, a different dyeing being used to differentiate each process. The yarns of the different processes, as well as untreated scoured yarn, were dyed together and then knitted up separately into socks of the specification given on p. 138.

The dyeing procedure used was the same for all material. In order to obtain the wash and light fastness demanded in shrink-resisted men's socks, premetallised acid dyes were employed.

Goods were entered at 50°c. into 40 vol. of liquor containing 1% Accozine (Ciba) (a mixture of pyridine bases and a sulphonated oil), 5% sulphuric acid, and 10% Glauber's salt, worked for 15 min., and lifted, and the previously dissolved dye was then added. The bath was raised to the boil in 40 min., 1% Neolan Salt IIa (Ciba) then added, and boiling continued for 90 min. The goods were rinsed, neutralised with 0·5% ammonia (26%), and rinsed again. The dyeings used were selected from the point of view of showing up any differences in depth between treated and untreated wool on the one hand, and between wool and nylon on the other—

B	...	0·5%	Neolan Blue 2R (Ciba)
C	...	0·5%	Neolan Blue GG
M	...	2%	Neolan Yellow BE
R	...	1·5%	Neolan Green BL conc.
		0·35%	Neolan Red BRE

It was found that the treated wool material dyed darker in every case except in Process C, Neolan Blue GG being, of course, the best levelling dye of the premetallised class. The greatest shade difference occurred with Process R. No true union shade was obtained on wool–nylon with any of the dyeings, the worst being, as expected, Process R

TABLE IV

No. of wearing cycles...	Handle in Washing					Colour Bleeding				
	1	2	3	4	5	1	2	3	4	5
B	Slimy	Slimy	Slimy	Slimy	Slimy	Slight	Slight	—	—	—
C	Very slimy	Very slimy	Very slimy	Very slimy	Slimy	Slight	Slight	Slight	—	—
M	Like B	Like B	Like B	Like B	Like B	—	—	—	—	—
R	Normal	Normal	Normal	Normal	Normal	Much	Much	Much	Slight	Slight

with the nylon pale green, and the best again Process C with Neolan Blue GG.

SERVICEABILITY

A panel of nine members was instructed to wear the socks produced in this experiment, each pair consisting of a treated and an untreated sock marked so that every day of wear right and left would be changed. Washing by hand closely approximating to domestic practice was carried out at eight regular intervals of 25 hours' wear (2 days with one reversal of sides). All socks were washed together at 40 vol. and 40°C. with 5 g. neutral soap flakes per litre for 15 min., the pH being 8·5 at the start and 7 at the end. Washing was followed by lukewarm rinsing and air drying flat on a table. During the wash test the treated and untreated socks were compared for colour bleeding and "sliminess", and after air drying the dimensional change of an 8-in. distance marked along the foot of each sock was measured. "Sliminess" and colour bleeding results are shown in Table IV.

The tests were not carried beyond the fifth wearing cycle, as there was then no significant change in behaviour. The apparently "normal" handle of R during washing is probably due to the harshness of the yarn counteracting sliminess due to swelling in alkaline solution. Colour bleeding was in every case stronger in the treated sock but not greatly different. Much bleeding of the green component took place in process R, the shade turning progressively duller.

TABLE V

Change in Length (in.) based on Original 8-in.

Wear Cycle (hr.)	B	Control	C	Control	M	Control	R	Control
25	–	–	−¼*	¼	–	¼	−¼	–
50	–	¼	−¼	¼	¼	¼	−¼	¼
75	–	¼	−¼	¼	¼	¼	–	¼
100	–	¼	−¼	¼	¼	¼	–	¼
125	–	¼	−¼	¼	¼	¼	–	¼
150	–	¼	−¼	¼	¼	¼	−¼	¼
175	–	¼	−¼	¼	¼	¼	–	¼
200	–	¼	−¼	¼	¼	¼	–	¼
After milling	¼	¼	–	1¼	¼	¼	−¼	¼

* Negative figures indicate stretching during wear.

At the end of the experiment socks were inspected for type and position of damage in wear and were then given a deliberate "overtreatment" consisting of "milling" for 20 min. in a gyrating washing machine in 2% soap at 60°C. and pH 9·5 followed by rinsing with warm water till the wool showed pH 7. Dimensional changes in inches based on the original length of 8 in. in each case are shown in Table V.

CONCLUSION

The results corroborate findings on considerable chemical modification in the cases of Processes C and R, which, under the conditions of the experiment, resulted in overtreatment. It is also evident that quite different shrinkages occur in the untreated material, depending on the individual wearer and his habits. Results obtained in Processes B and M would indicate adequate shrink-resistance without excessive chemical or mechanical damage, a compromise typical of this kind of textile finishing.

It remains to reconcile the high strength and extensibility conferred on the yarn by Process R with the high degree of chemical damage and poor wearing qualities. Whilst all other socks remained intact during the 200 hr. of wear, the R socks showed rapid wear with holes occurring after 120 hr. in the parts subjected to greatest abrasion. In this connection it is noted that the nylon reinforcement also acts as an abradant. That the R-treated wool had undergone a process of "embrittlement" with lowered resistance to abrasion and flexural fatigue is indicated not only by short service life, despite great tensile strength, but also by the fact that the yarn showed a tendency to break during knitting, whilst the other yarns were satisfactory.

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Some Observations on the Hardening Process used in Felt Manufacture I—Factors influencing Hardening*

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The hardening of masses of loose wool under various conditions is studied, the effectiveness of the operation being assessed by measuring the air permeability (on a Cambridge instrument) and the breaking load of samples of the hardened material. It is shown that on a Garson plate hardener maximum hardening is effected by processing for 80 sec., prolonged treatment yielding weaker hardened pads. The use of high steam pressures also tends to produce weak pads. Hardening is more effective in presence of acids than under neutral or alkaline conditions, but if the amount of sulphuric acid exceeds about 2%, degradation occurs. Phosphoric, acetic, and formic acids behave similarly to sulphuric acid at the same pH value, but if the pH is less than 2 the strength of the hardened pad falls. Chlorinated wool is more difficult to harden than unchlorinated wool.

The felting operations employed in the non-woven felt industry may be divided into two—(i) hardening, in which a mass of loose fibres, after being roughly shaped by carding and forming, is subjected to a high-speed vibratory motion in presence of steam while under considerable mechanical pressure; and (ii) fulling, in which the hardened material is felted further in aqueous media at comparatively low temperatures. The latter is analogous to the acid milling used in cloth finishing, and the theory which has been developed[1] in this connection may well be expected to apply. Hardening, however, is carried out under very different conditions, and it is possible that different principles may be involved. Judging from the lack of published data, the process has not previously been studied systematically, and the present series of papers is an account of experimental work carried out to discover the factors influencing hardening, and to provide data upon which a theory of hardening could be based. It was first necessary to find a method of hardening which yielded reproducible results and to devise a method of describing the physical properties of the hardened products. Measurements of tensile strength and air permeability have been found useful in this connection, and it has thus been possible to study the hardening of wool under various conditions. To explain the results so obtained in terms of single-fibre properties it was found necessary to measure certain physical characteristics under conditions closely allied to those used in hardening. This work will be described in Part II of the series, while in Part III an account will be given of some experiments on the hardening of blends of wool and certain non-felting materials.

Experimental

PRODUCTION OF HARDENED PADS

After many preliminary experiments in which small-scale apparatus was used, it was decided that the results obtained were less reliable than those obtained on a commercial hardening machine, provided sufficient care was taken in preparing the pads. The experimental details are as follows—

CARDING— In all the experimental work, a web of fibres was prepared by carding wool on a single-cylinder carding engine of the type used in hat manufacture, the web being wound at full width on a large drum 53 cm. in diameter. On account of the small quantities of wool used in most of the trials, the weighing device at the feed end of the card was unsuitable, and the wool was therefore placed evenly on the feed sheet by hand, being well opened before use. To prepare a pad, a certain weight (usually 750 g.) of wool was fed into the machine. As much as possible of this wool was collected on the drum, and by suitable trimming it was possible to obtain a uniform lap 40 cm. wide and of length equivalent to the circumference of the drum. Laps of this length were cut into two equal parts. When dealing with treated wools, the machine was allowed to run for at least 15 min. to clean it before introducing the test sample. A small amount of the treated wool was then passed through the machine, and the web formed was rejected. The bulk of the material was then carded, it being found that in this way contamination from previous cardings was avoided. It was interesting to observe that wools which had received different pretreatments behaved differently during carding. For example, wools treated with acids carded more easily than wools which were neutral or alkaline.

HARDENING— To harden the pads of carded wool a Garson plate hardening machine was used. It was of the usual commercial design, and consisted essentially of a perforated metal table, through which steam was allowed to pass, and an upper plate of area similar to that of the top of the table. The plate could be oscillated very rapidly, being controlled by two eccentrics and hinged along one of its sides. When the upper plate was lowered into contact with the top of the table, the web, which was placed between the two surfaces, was subjected to considerable pressure by the action of two struts which operated on the plate through two stout metal springs. Both the upper and lower plates were covered with fabric.

Before use the machine was warmed by allowing steam to pass freely through it for about 15 min. The upper rubbing unit was raised, a pad of wool placed on the lower plate, and the upper plate brought down on to the lower one, the struts being placed in position. The steam valve was opened, and steam allowed to pass through the wool for 1 min. During all the experiments the rate of steaming was maintained constant. After 1 min.

* The substance of this paper formed part of lectures entitled *The Felting of Animal Hairs* given to the Manchester Section of the Society in Stockport on 6th February 1947, Mr. N. HAMER in the chair, and to the Midlands Section in Nuneaton on 8th November 1950, Mr. W. L. LANGTON in the chair.

steaming the upper plate was set in motion and allowed to run for a fixed time. To compensate for any differences in the work performed on the wool in various parts of the machine, it was stopped after half the total hardening time had expired, and the partly hardened pad removed, turned over and through 180°, and given a further and exactly similar hardening treatment. In much of the work 60 sec. hardening in each position was given. From the large pieces of "felt" produced in this way it was possible to cut uniform portions of at least 30 cm. × 90 cm.

The nature of the covering used on the hardening plates proved to be important, and both plates were covered with good-quality linen duck backed with a coarse jute fabric. None of the results quoted in the following sections was obtained immediately after the plates had been re-covered, as new coverings were found to yield variable pads. In all cases sufficient wool was processed to stabilise the coverings before test samples were hardened, and, as far as could be determined, any further changes which took place had no effect on the hardening. The machine was set so as to give the upper plate a reciprocating motion of amplitude 0·5 cm. The frequency of vibration was checked at intervals, and only occasionally was it outside the limits 1080–1100 vibrations per minute.

METHODS OF TESTING

The pieces of felt obtained from the hardening machine were conditioned in a room maintained at 22·2°C. and 65% R.H. prior to making measurements of breaking load or air permeability.

(1) Breaking Load—These determinations were made on the F.A.C.O.N. upright machine, a constant-rate-of-traverse instrument in which the rate of extension of the sample was 18 cm. per minute. Samples to be tested were cut by means of a die to the dimensions shown in Fig. 1, twenty-four test samples being taken from each large piece

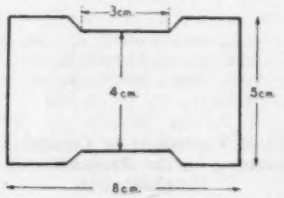
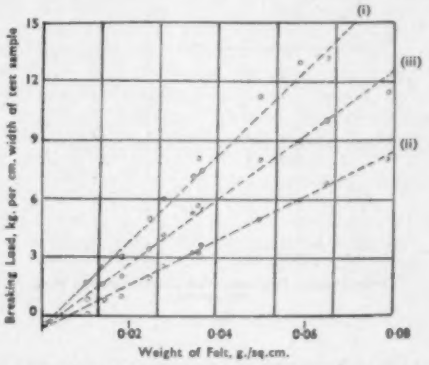


Fig. 1

of felt; half were cut with the length of the sample parallel to the length of the hardened pad, i.e. in the direction of the main fibre orientation, and half in a direction perpendicular to the length. The results are expressed in terms of grams per centimetre width of test sample.

Clearly the breaking load of a hardened pad will depend on the weight of wool in the pad, and as it was found to be nearly impossible to produce carded webs of exactly the same weights, a series of experiments was carried out in which a range of webs of various weights was prepared and hardened, and their breaking loads were measured as described previously. The results are illustrated graphically in Fig. 2. There is a straight-line



(i) Breaking load measured parallel to direction of fibre orientation
(ii) Breaking load measured perpendicular to direction of fibre orientation
(iii) Mean value

Fig. 2

relationship between the breaking load and the weight of the felt (per unit area) over the range investigated. The tensile property of any web may therefore be expressed by the ratio—

$$\frac{\text{Breaking load per unit width}}{\text{Weight of felt per unit area}}$$

The lines do not, however, pass through the origin but meet at another point on the strength axis. An empirical correction was therefore introduced to allow for this experimental finding and in all cases the tensile properties of the hardened pads used in the present work are expressed by the term—

$$\frac{\text{Measured breaking load per unit width of test sample} + 600}{\text{Weight of felt per unit area}}$$

$$\left(\frac{\text{g./cm.}}{\text{g./sq. cm.}} = \text{cm.}\right)$$

This is described as the *strength ratio*.

There was considerable difference between the strengths of samples cut parallel to the direction of main fibre orientation and those cut perpendicular to this direction, as shown by the following figures obtained on one piece of felt—

	Strength Ratio (cm.)
Parallel to main fibre orientation...	$1{\cdot}92 \pm 0{\cdot}035 \times 10^4$
Perpendicular to main fibre orientation	$1{\cdot}05 \pm 0{\cdot}02 \times 10^4$

It was therefore decided to take the mean of the two sets of measurements and to use this value as an expression of the strength of the pad. Values of the strength ratio for measurements taken at various angles to the direction of the main fibre orientation will be intermediate between those given above, as indicated in Fig. 3. It is interesting to note that the curve is not a straight line.

(2) Air Permeability—For the present work the apparatus made by the Cambridge Instrument Co. was used, in which the pressure required to suck

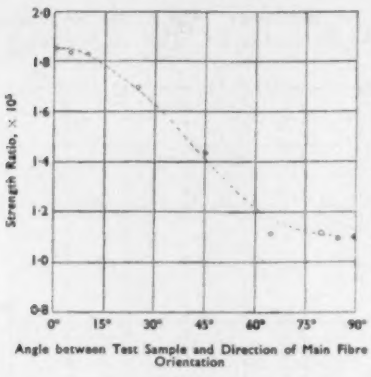


FIG. 3

air through a sample of known area (64·5 sq.cm.) at a constant rate of 7·33 c.c./sq.cm./sec. is measured, so that the results are inversely proportional to the true permeabilities. Twenty measurements were made to obtain a reliable mean. As with measurements of strength, it was necessary to apply corrections for the varying weights of the test samples. The results of determinations of the permeabilities of felts of various weights (per unit area) are shown in Fig. 4, from which it is evident

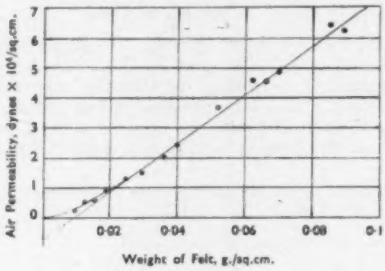


FIG. 4

that, within the range encountered in most of the experiments in the present work, there is a linear relationship between air permeability and weight of felt per unit area. Extrapolation of this line cuts the vertical axis at — 8000 dynes/sq.cm., and hence in all subsequent work the term *air permeability ratio* was used, this quantity being—

$$\frac{\text{Measured air permeability} + \frac{8000}{}}{\text{Weight of felt per unit area}} \quad \left(\frac{\text{dynes/sq.cm.}}{\text{g./sq.cm.}} = \frac{\text{dynes}}{\text{g.}}\right)$$

Determinations of air permeability ratio on several samples of hardened web indicated that, if sixteen tests were made, the value could be relied upon to $\pm 3\%$.

General considerations suggest a relationship between air permeability and the fibre : space ratio of the material, and examination of some of the felts used in the present work revealed a linear relation between permeability and apparent density

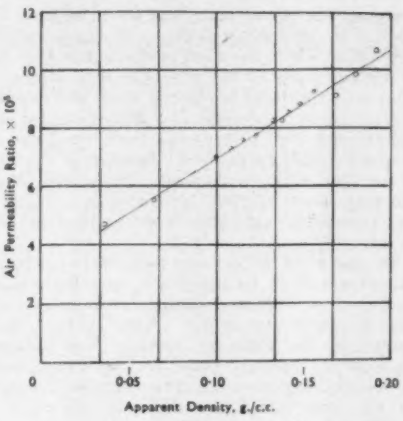


FIG. 5

of the material within the range of densities 0·05–0·20 g./c.c. (Fig. 5). This is a useful observation, for it indicates that the permeability ratio gives a measure of the air space within the boundaries of the material, and such measurements may therefore be substituted for the more difficult determinations of bulk density.

MATERIAL USED

Although blends consisting largely of noils are used in the felt trade, these were not considered to be suitable for the present work, as it is difficult to standardise their quality. 64s Australian merino wool—good felting material of reasonably long staple, which may be readily obtained and repeated—was therefore used. The material was scoured in a four-bowl set under the conditions summarised below, after which it was dried and allowed to condition—

Bowl		
1st	...	0·5% Soap + 0·07% soda ash at 54·5°C.
2nd	...	0·5% Soap + 0·07% soda ash at 53°C.
3rd	...	0·5% Soap + 0·07% soda ash at 52°C.
4th	...	Water

Effect of Variations in Conditions of Hardening on the Properties of the Resulting Felt

Factors which influence the quality of the hardened felt include duration of hardening, steam pressure, presence of acid or alkali in the wool, and composition of the blend. In the present work the properties of pads made under various conditions have been measured to find the effect of altering some of these factors.

TIME OF HARDENING

A number of pads of carded wool of approximately equal weight were prepared and hardened for various times. Steam pressure and rate of oscillation of the plate of the machine were maintained constant at 5 lb./sq. in. and 1100 vibrations per minute respectively. Measurements of the air

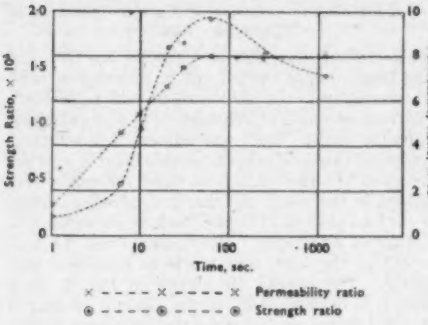


FIG. 6

permeability and the tensile strength were subsequently made on the hardened material, the results obtained being shown graphically in Fig. 6, in which permeability ratio and strength ratio are plotted against the logarithm of the time of hardening. This refers to the total time of hardening, all samples being turned half way through the operation as described earlier.

The shapes of the two curves are interesting. Both show that wool hardens very rapidly in the early stages and that maximum hardening is obtained after 80 sec. in the machine under the conditions chosen. As the time is increased further, the strength ratio falls quite markedly, but the permeability ratio remains more or less constant. The fall in strength ratio is substantial, as much as 25% of the maximum strength being lost if the time of hardening is prolonged to 1200 sec. This is no doubt due to degradation of the wool by the steam, and emphasises the danger of prolonged hardening.

STEAM PRESSURE

In these experiments masses of wool were hardened for a fixed time, but the pressure of the steam used to facilitate the process was altered in each case. Care was taken to heat the machine to the same temperature as the steam before commencing hardening, and the pressure of the steam was measured just before it entered the machine. In all the experiments the time of hardening was 120 sec. and the upper plate of the hardener was made to vibrate at 1100 vibrations per minute. Measurements of breaking load and air permeability were made on the hardened pads as described previously.

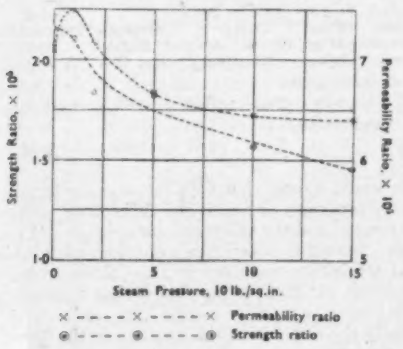


FIG. 7

It is seen from Fig. 7 that an excessively high pressure reduces the efficiency of the hardening process. There is a continuous fall in the strength ratio as the rate of steaming is increased, the fall being most marked in the early stages. The same general effect is noted on the air permeability curve, but in this case the fall occurs after an initial rise. The graphs indicate quite clearly an optimum steaming pressure for hardening. It is impossible to define the pressure exactly, for the pressure within the steam chamber is unknown and varies with the design and covering of the machine. It should, however, be kept as low as possible to minimise the attack on the wool, which is no doubt partly responsible for the fall indicated in the graphs.

ACIDITY OR ALKALINITY OF THE WOOL

It is well known that the ease of felting wool cloths is greatly affected by the pH under which the operation is carried out, acids in particular facilitating the process. Experiments were therefore carried out to determine the effect of pH on hardening. In general the experiments consisted in soaking a suitable amount of wool (1000 g.) in an acidic or alkaline solution (10 litres) for 18–24 hr., hardening the treated wool after it had been centrifuged, opened and conditioned in the laboratory, and finally measuring the permeability and breaking load of the hardened pads. The pH of the residual liquor was measured with a Cambridge Instrument Co. glass electrode, and in the cases of wool containing considerable amounts of acid the acidity was measured by the pyridine extraction method[2]. For these determinations 2 g. of wool, conditioned at 65% R.H. and 22·2°C., was placed in a conical flask, and 200 ml. of a 0·5% solution of pyridine added to the wool. After 24 hr., two 50-ml. samples of the liquor were withdrawn and titrated with 0·01 N. caustic soda solution, using phenolphthalein as indicator. The tests were done in duplicate. Three series of experiments were carried out—

I—Hardening at Various pH Values

Several batches of wool were immersed in various acid or alkaline solutions, the pH being measured before and after treatment, after which the wool was centrifuged, air-dried, conditioned, and hardened in the usual way. Details of the solutions used are summarised in Table I, and the results of measurements of the air permeability and tensile strength ratios are shown in Fig. 8.

It is at about pH 4·8 that wool begins to take up acid, and at this pH value both curves begin to rise sharply with fall in pH, a maximum being reached at pH 2. Values of the air permeability ratio, which expresses the consolidation of the material, then remain almost constant as the pH

TABLE I

Composition of Solution used for soaking Wool	Equilibrium pH of Bath
1·9 N. Sulphuric acid	0·1
0·9 N. Sulphuric acid	1·1
0·2 N. Sulphuric acid	1·2
0·15 N. Sulphuric acid	1·4
0·09 N. Sulphuric acid	2·0
0·03 N. Sulphuric acid	3·2
Distilled water	5·2
0·1 N. Boric acid	7·5
0·1 N. Borax	9·4
0·1 N. Sodium carbonate ...	10·1
0·1 N. Caustic soda	12·7

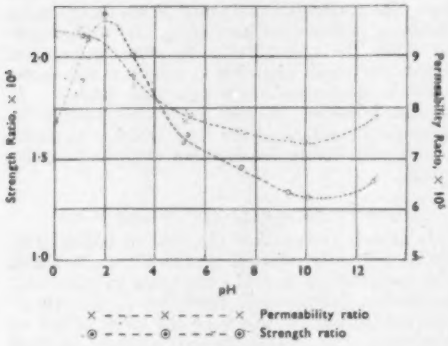


FIG. 8

falls still lower, whereas the strength ratio falls very considerably. On the alkaline side of the isoelectric point of wool the results show that both characteristics fall as the pH is raised. The effect is much more marked in the case of strength measurements, which seems to indicate that the fall in strength ratio is largely due to fibre degradation. Steaming under strongly alkaline conditions certainly leads to considerable degradation, for the discoloration of the felt increases as the pH rises. The felt hardened under the most alkaline conditions was quite yellow.

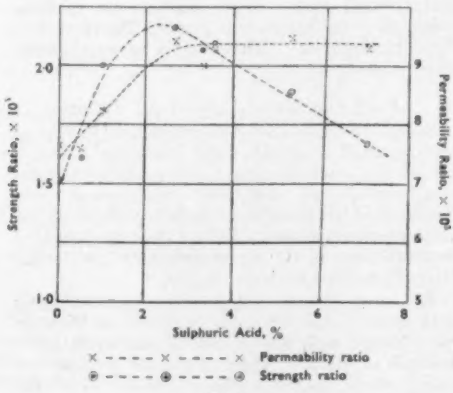


FIG. 9

II—Hardening in presence of Various Amounts of Sulphuric Acid

It is evident that the best results are obtained when hardening is carried out in presence of acid, and consequently the hardening of wool in presence of various amounts of sulphuric acid was examined in greater detail. Wool was immersed in solutions of sulphuric acid of various concentrations, and the properties of pads made from these materials were assessed in the usual way, hardening being carried out for two periods of 60 sec. each using steam at a pressure of 5 lb./sq.in. Measurements of the acid content of the wool were made as described previously. The results are shown in Fig. 9, from which it is evident that, as the amount of acid in the wool increases, so does the efficiency of the hardening process, the greatest effect being obtained on wool containing about 2% of acid (calculated on the weight of the wool). If the concentration of acid is increased further, the strength ratio of the hardened material falls, a decrease of some 20% being recorded in the sample containing the most acid. The curve plotted from air permeability data also shows a sharp increase in consolidation as the amount of acid in the wool rises to 2%, and wools containing more acid give this same limiting value. The fall in strength ratio may be explained either by a reduction in the interlacing of the fibres, or by a weakening of the fibres due to the action of the acid, and in view of the constant value shown on the permeability curve it is probable that the latter cause is the more important. The degradation is no doubt due to hydrolysis of the main chains by the acid at high temperatures.

Whilst considering the effect of sulphuric acid on hardening, a trial was carried out to assess the effect of carbonising on the ability of wool to be hardened. A batch of wool was carbonised by allowing it to come to equilibrium in a solution of sulphuric acid (initially 0·19 N.), centrifuged, and then heated in an oven at 104°C. for 30 min. It was then rinsed in water, air-dried, and hardened as previously described. The results obtained on samples of this felt and a control sample of the same wool treated with acid as above but allowed to dry in the air are given in Table II.

TABLE II

Initial Concn. of Acid (N.)	Equilibrium pH of Bath	Acid on Wool (%) Before Hardening	Acid on Wool (%) After Hardening	Strength Ratio (× 10³)	Air Permeability Ratio (× 10⁴)
UNCARBONISED					
1·9	0·14	7·05	6·94	1·68	9·38
CARBONISED					
1·9	0·14	6·65	6·49	1·40	8·70

It would appear that both ratios are reduced by the carbonising, although the differences between the treated and the untreated material are not very large. Since the carbonising conditions used in this trial are rather more severe than those normally employed, it must be concluded that commercial carbonising, carefully carried out, reduces only slightly the response of the material to the hardening process.

III—Effect of Acids other than Sulphuric

Similar experiments were carried out on wool which had been soaked in solutions of phosphoric, acetic, or formic acid, measurements of air permeability and tensile strength being made on the hardened products. The figures shown in Tables II and III indicate that all the reagents yield similar results. Phosphoric acid behaves very much like sulphuric, and at low pH values gives rise to degradation, which results in a fall in strength. Formic and acetic acids do not show this effect, owing no doubt to the difficulty of realising a sufficiently low pH. Even when using solutions of 2 N. acid the amount adsorbed was not greater than 1·3%. Moreover, these weaker acids are also volatile in steam, and more acid is therefore lost during hardening. As much as 30% of formic, and 60% of acetic, acid may be removed from the wool during the operation. It appears from these results that the effect of acids in facilitating hardening is largely due to the low pH.

TABLE III

Initial Concn. of Acid (N.)	Equilibrium pH of Bath	Acid on Wool (%) Before Hardening	Acid on Wool (%) After Hardening	Strength Ratio ($\times 10^3$)	Air Permeability Ratio ($\times 10^3$)
Phosphoric Acid					
0·06	4·05	0·63	0·60	1·83	8·35
0·31	2·04	1·49	1·53	1·83	8·14
1·9	1·22	7·94	7·98	1·80	8·39
6·1	0·83	12·93	12·70	1·59	8·10
Formic Acid					
0·04	3·80	0·39	0·34	1·86	8·46
0·13	3·31	0·71	0·55	1·81	8·14
0·13	3·51	0·80	0·70	1·81	8·87
0·63	2·50	1·14	0·76	1·90	8·74
1·6	2·34	1·30	0·77	1·90	8·18
2·5	2·00	1·28	0·84	1·93	8·64
Acetic Acid					
0·04	4·50	0·21	0·18	1·79	8·06
0·10	3·94	0·34	0·25	1·74	7·85
0·53	3·27	0·63	0·32	1·83	8·11
1·2	2·55	1·19	0·44	1·77	7·83

HARDENING OF CHLORINATED WOOL

In studies of fabric shrinkage and felting the importance of the surface scales of the fibres is clearly recognised, and it is likely that these morphological components influence the results obtained during hardening. To find out whether this is in fact the case, a series of experiments was carried out on samples of wool which had been chlorinated to modify the surface properties. The method of chlorination is illustrated by the following example—

Wool (3000 g.) was thoroughly wetted out in water, and then centrifuged before being placed in 110 litres of water to which had been added 615 ml. of concentrated sodium hypochlorite solution (9·67% available chlorine) and 250 ml. of glacial acetic acid. Treatment was allowed to continue for 45 min., the liquid being agitated at frequent intervals. The wool was then removed from the solution and thoroughly centrifuged. Analysis of the residual liquor showed that 1·9% of chlorine (on the weight of the wool) had been adsorbed and the final pH was 4·1. The wool was then given an antichlor treatment by immersing it for 5 min. in a solution prepared by dissolving 170 g. of anhydrous sodium bisulphite in 110 litres of water, after which it was thoroughly washed in running water. The final pH was 3·76. The wool was then divided into two lots, and each was adjusted to a different pH by immersion in dilute sulphuric acid solution and in 1% sodium borate, and in this way three samples of pH values 1·6, 4·7, and 9·0 respectively were obtained.

Employing a similar chlorination technique samples of more severely chlorinated wool were also prepared. The chlorinating bath was made by mixing 1840 ml. of sodium hypochlorite and 450 ml. of acetic acid, and this enabled 6·0% of chlorine to be adsorbed. The final pH was 4·02. Samples of this material were brought to equilibrium in solutions of 0·1 N. sulphuric acid or 0·1 N. sodium borate, as described before, the final pH values being 1·4 and 8·8 respectively.

Each sample of chlorinated wool was carded, hardened, and tested as described previously.

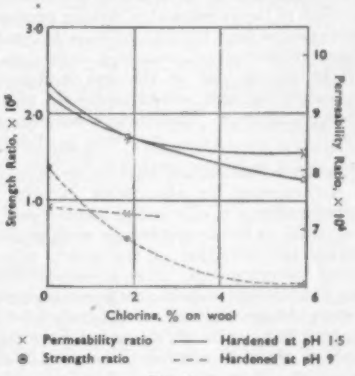


FIG. 10

It will be seen from Fig. 10 that, as the quantity of chlorine used in the chlorination increases, the strength of the hardened material falls considerably, this being particularly pronounced with wool which has been hardened in an alkaline condition. The air permeability is affected to a lesser extent, and even the felt formed by hardening wool chlorinated with 6·0% of chlorine and subsequently made alkaline was found to have a permeability not very different from that of unchlorinated wool hardened under similar conditions. The value shown in this case is not a precise one, as difficulties in carding the material gave rise to an uneven felt. Since chlorination reduces the differential frictional effect (D.F.E.) of the wool[3], the above results suggest that, if this characteristic is eliminated, hardening, i.e. the development of strength in the web following appropriate mechanical treatment, is more difficult. Decreases in D.F.E. cause decreases in

effectiveness of hardening. Chlorination, however, has less effect on the tightness of the interlacing as revealed by the results of permeability measurements. A more detailed consideration of the importance of D.F.E. in relation to hardening will be made in a subsequent paper.

* * *

The authors have pleasure in acknowledging the financial assistance given by Messrs. S. Hubbard & Co. Ltd., of Luton, which made it possible for this work to be carried out.

Department of Textile Industries
University of Leeds

(Received 25th September 1950)

References

[1] Speakman, Stott, and Chang, *J. Textile Inst.*, **24**, T 273 (1933).
[2] Barritt, *ibid.*, **26**, T 87 (1935).
[3] Mercer, *Nature*, **155**, 573 (1945).

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

The Absorption of a Direct Cotton Dye Anion associated with Various Cations

The Editor

Sir,

The preparation of the pure water-soluble sulphonic acids of the direct dyes by an ion-exchange technique [1] has made readily available salts other than those of the common alkali metals. This has prompted an exploratory reinvestigation of the effect of the cation in the dyeing process. It is to be expected from the Law of Mass Action that, for higher concentrations of added electrolyte, the use of the sodium salt of the dye, and not the corresponding dye salt, would have little effect on the dyeing behaviour. This has now been shown to be true.

It has been established that in general there exists an increase in absorption of dye with increasing valency of the added electrolyte [2], but there appears to be no systematic work published concerning the variation of the cation within a particular valency group. It is, moreover, difficult to draw any definite conclusions from much earlier work since at times salts of several acids have been chosen for examination which cannot have given a fixed pH in the dye solution between each set of experiments. It would seem, therefore, that no definite conclusions have yet been reached as to whether at a fixed pH similar ions of the same valency, under identical dyeing conditions, would give dyeing to the same equilibrium value. In our experiments the pure dye acid was neutralised with a series of organic and inorganic bases to give the corresponding neutral dye salts.

The equilibrium dye absorption for the purified Sky Blue FF (*C.I.* 518) anion associated with different cations in the presence of the corresponding chloride at pH 7 $\pm$ 0·5 unit are shown in the accompanying table.

The results indicate that there is a definite variation in equilibrium absorption for the various cations. They do not, therefore, confirm in detail the mathematical theory of dye absorption of Willis *et al.* [3] that salts of the same valency should dye the same equilibrium absorption. This is probably due both to the difficulty of knowing the thermodynamic activities of the dye and added electrolyte, especially in the cellulosic phase [4], and to steric effects. In particular, conductivity experiments (see table) suggest that, *within groups of similar cations*, there exists a relationship between the size of the solvated ion and the extent of absorption; the larger the ion the lower the absorption.

Absorption of Sky Blue FF Anion on 150/27 Viscose Rayon

(0·00101 M. dye, 0·0214 N-BCl (stoichiometrically equivalent to 5·0% NaCl), 90°C. in 40 vol.)

Cation (B)	Equilibrium Absorption (%)	Ion Conductance of Cation
Ammonium	86	65
Potassium	84	65
Sodium	81	43
Lithium	76	33
Tetramethylammonium ...	72	65
Trimethylbenzylammonium ...	68	32·5
Tetraethylammonium ...	60	43
Triethylbenzylammonium ...	57	22·5

Comparison of the dyeing results corrected for variable equilibrium absorption shows that there is no great variation in the diffusion constant for the systems examined. This would be expected, since the diffusion at fixed ionic environment should be dependent only upon the largest ion present, viz. the common dye anion.

This work is being extended with a consideration of the effects of temperature, concentration, and fibre structure.

Yours faithfully
H. B. Mann
R. W. Richardson

Courtaulds Ltd.
Bocking
Braintree
Essex

16th February 1951

[1] Richardson, *Nature*, **164**, 916 (1949).
[2] Vickerstaff, *The Physical Chemistry of Dyeing* (London: Oliver & Boyd 1950), p. 224.
[3] Willis, Warwicker, Standing, and Urquhart, *Trans. Faraday Soc.*, **41**, 506 (1945).
[4] Standing, Private communication.

Notes

Meetings of Council and Committees March

Council— No meeting
Publications— 20th
Colour Index Editorial Panel— 6th
Fastness Tests Co-ordinating— 13th
Terms and Definitions— 8th
Reviews of Textile Progress— 16th
1951 Conference— 2nd
Leather Dyes— 2nd

The Chemical Society Research Fund

The Research Fund of the Chemical Society provides grants for the assistance of research in all branches of Chemistry, about £700 per annum being available for this purpose. Applications for grants should be submitted not later than Tuesday, 1st May 1951. Forms of application together with the regulations governing the award of grants may be obtained from the General Secretary, The Chemical Society, Burlington House, Piccadilly, London W.1.

Manchester Municipal College of Technology Scholarships and Exhibitions

The Governing Body offer six scholarships and six exhibitions varying in value up to a maximum of £65 plus approved fees if the student is resident at home, and £140 plus approved fees if resident in hostel or lodgings, to part-time Manchester students and others, tenable in any one of the full-time day courses of the College for three academic years, on condition that persons holding these scholarships prepare themselves for the degree of Bachelor of Technical Science (B.Sc.Tech.). Forms of application may be obtained from the Registrar, College of Technology, Manchester 1, and completed forms must reach him not later than 1st June 1951.

Chemistry and Physics of Textiles and Dyeing

An advanced course of ten lectures will be given at Bradford Technical College on Wednesdays at 7 p.m., beginning on 2nd May 1951. Two lectures will be devoted to each of the following topics— measurement of colour, metal–dye complexes, action of light on dyes and fibres, fibre–liquid interaction (swelling and solution), and kinetics of dyeing (wool and cotton). Application forms, obtainable from the Principal of the College, should be returned as soon as possible.

Educational Travel to U.S.A.

Youth Travel Ships Ltd., a non-profitmaking organisation, is planning travel to and from U.S.A. this summer. The cost by sea will be £50–65 return, and accommodation will be on Y.H.A. lines, with lectures and recreation. Return fare by air is £86. Those without dollars may be able to get brief seasonal work near New York to cover expenses in U.S.A. Further particulars can be obtained from the Acting Secretary, Youth Travel Ships Ltd., 7 New Square, London W.C.2.

OBITUARY NOTICE

William Rearden Atkin

We regret to record the death at the age of 60 of W. R. Atkin, Professor of Leather Industries in the University of Leeds. The whole of Professor Atkin's working life— excepting a period of distinguished service in the 1914–18 war— was spent in the University, where he had been trained first in pure chemistry and then in the chemistry of leather manufacture under the late Professor Procter. He made many important contributions to the science of leather manufacture, his investigations on a wide range of topics gaining world-wide recognition. Much of his work found direct application in industry, and his opinions on technical matters were greatly valued. Professor Atkin was also a great teacher, who rapidly gained the affection and co-operation of his students, and because of his long connection with the University, first as lecturer and later as Professor, he was able to influence a large number of men, many of whom now hold responsible positions in the leather trade. His lectures outside the University were always stimulating, and the occasions when he addressed Sections of the Society were no exception. He was able to present involved subjects in a lucid and interesting manner and to establish an intimate relationship with his audience. This friendliness was, perhaps, his most characteristic feature, and many members of this Society, especially those concerned with the colouring or processing of leather, remember with pleasure a man who was not only a first-class scientist and teacher, but also a genial Yorkshireman. C. S. WHEWELL

New Books and Publications

Review of Textile Progress Volume I 1949

Compiled and published jointly by the Textile Institute and the Society of Dyers and Colourists. Pp. 403. Price, 20s. 0d.

For many years workers in the field of textile chemistry have been indebted to the Society of Chemical Industry for an annual review of their subject in the Society's *Reports on the Progress of Applied Chemistry*. These reviews have always

maintained a high standard, but, since the last war, the bulk of material to be surveyed has become so large that a separate volume is now required to deal with it adequately. In consequence, the Textile Institute and the Society of Dyers and Colourists decided to publish their own annual review, and the first volume, for 1949, has now appeared.

A glance at the list of contributors is sufficient to assure the reader of the uniformly high standard of this review, which covers the physics and chemistry of fibrous materials, fibre production, the production of yarns and fabrics, colouring matters, dyeing and printing, finishing, analysis and testing, laundering and dry cleaning, and building and engineering. The field covered is thus considerably wider than that surveyed in the relevant chapters of *Reports on the Progress of Applied Chemistry*.

As this volume is the first of the series, the contributors have not adhered strictly to material published in 1949, but have provided a suitable background. By providing sufficient space and allowing a certain amount of overlapping, the editors have enabled contributors to present their subject in a form which is easy to read and is not unduly compressed. The interesting section on colouring matters, however, is comparatively brief, and is directed to the dye-user rather than to the manufacturer of dyes.

The Committee concerned with the production of this volume have made an excellent beginning and deserve every encouragement. Their work is an important contribution to the welfare of the textile industry.

C. L. BIRD

A.S.T.M. Standards on Textile Materials
(with related information)

Prepared by A.S.T.M. Committee D-13 on Textile Materials. Pp. xii + 572. Philadelphia: American Society for Testing Materials. 1950 (issued annually). Price, paper, $4.50.

The A.S.T.M. standards on textile materials are well known to readers of the *Journal*, and the annual publication of these standards in their latest form ensures that the public are informed of new specifications and of changes in older ones. The present volume, published in October 1950, includes some ninety standards and "tentatives" covering a wide range of topics. New standards include methods of determining vegetable matter in scoured wool, snag resistance of hosiery, tests on bonded fabrics, magnetic rating of asbestos used for electrical purposes, and resistance of pile floor coverings to damage caused by insect pests. Some twenty-three older standards have been revised. As usual, the appendixes make interesting reading, as they contain recommendations on topics as widely divergent as tests for evaluation of properties related to the handle of soft-finished woven fabrics and an accelerated ageing test.

It is unnecessary to stress the valuable work carried out by Committee D-13, which is responsible for the formulation of the standards, and all who are interested in any aspects of textile testing should make certain that they are acquainted with the contents of the present volume.

C. S. WHEWELL

Introduction to Textile Chemistry

By B. E. Hartsuch. Pp. ix + 413. New York: John Wiley & Sons Inc. London: Chapman & Hall Ltd. 1950. Price, $4.75 or 38s. 0d.

During the last twenty years there has been a growing interest in textile chemistry, and the subject is now a well established part of the curriculum in textile departments of technical colleges and universities. There is, however, a dearth of good textbooks. Such books fall into two categories, the monograph for the specialist with a sound scientific background, and the more general textbook intended to give technologists and the more elementary students an introduction to the applications of chemistry in the textile industry. The book under review is of the second type and has been written primarily as a textbook for students. Consequently the difficulties of the average textile student are recognised, and the first two chapters are devoted to a lucid treatment of some of the common calculations used in chemistry and of elementary organic chemistry. These are followed by accounts of the chemistry of soap, water, and cellulose and its derivatives, and of the various textile fibres— cotton, wool, silk, rayon, nylon, man-made polymerised fibres, and man-made protein fibres. There is also a glossary of terms used in modern textile chemistry. The treatment in most cases is quite detailed. For example, under the heading "Wool" are discussed the morphological structure of the fibre, physical and chemical properties, raw wool scouring, felting, setting and shrinkproofing, and the reaction of the fibres with water, acids, alkalis, reducing and oxidising agents, and cross-linking agents.

The subject matter is well presented and is clearly explained, but a reasonable knowledge of pure chemistry is necessary to obtain the maximum benefit from the book. Although the first two chapters are designed to refresh the memories of technological students who have neglected their chemistry for a time, they do not form an adequate background for the study of the rest of the work. In many ways it would have been better to omit them and to devote the space to other aspects of textile science which are not considered. Many readers will regret the decision of the author to omit all consideration of dyes and dyeing, for although dyeing is often considered to be quite apart from the rest of textile processing, it is really only one operation in the larger scheme of converting fibres into finished cloth. Alternatively, the space could have been devoted to short accounts of such topics as surface tension, wetting, pH, titration curves, and the Donnan equilibrium. This would have enabled more emphasis to be laid on the physical aspects of textile chemistry. Nevertheless, the book is a notable contribution to the literature on textile chemistry and will be invaluable to students and teachers.

C. S. WHEWELL

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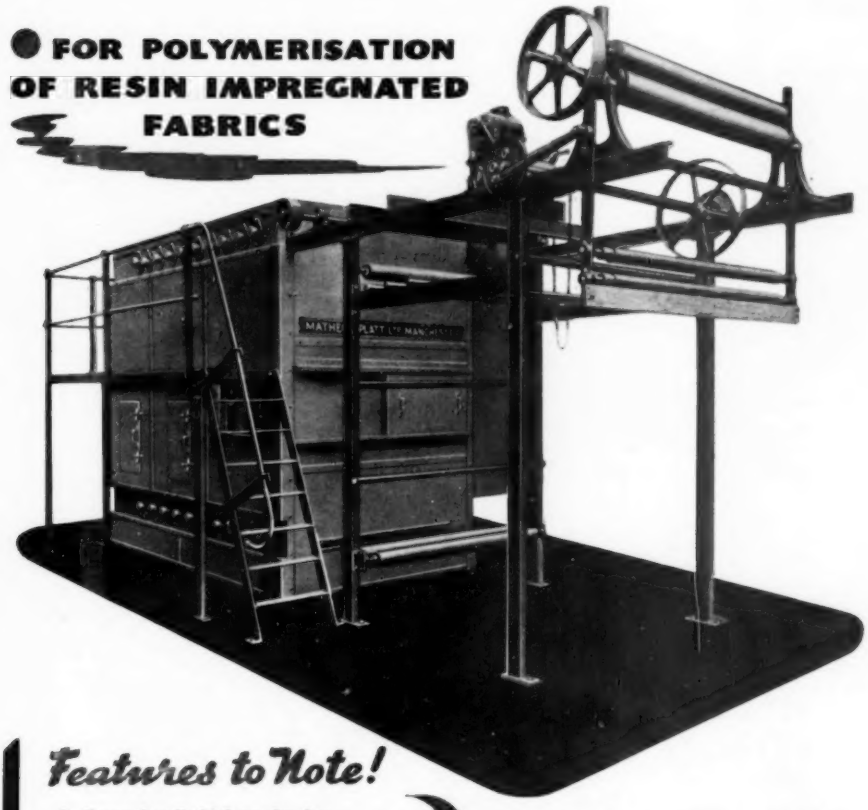

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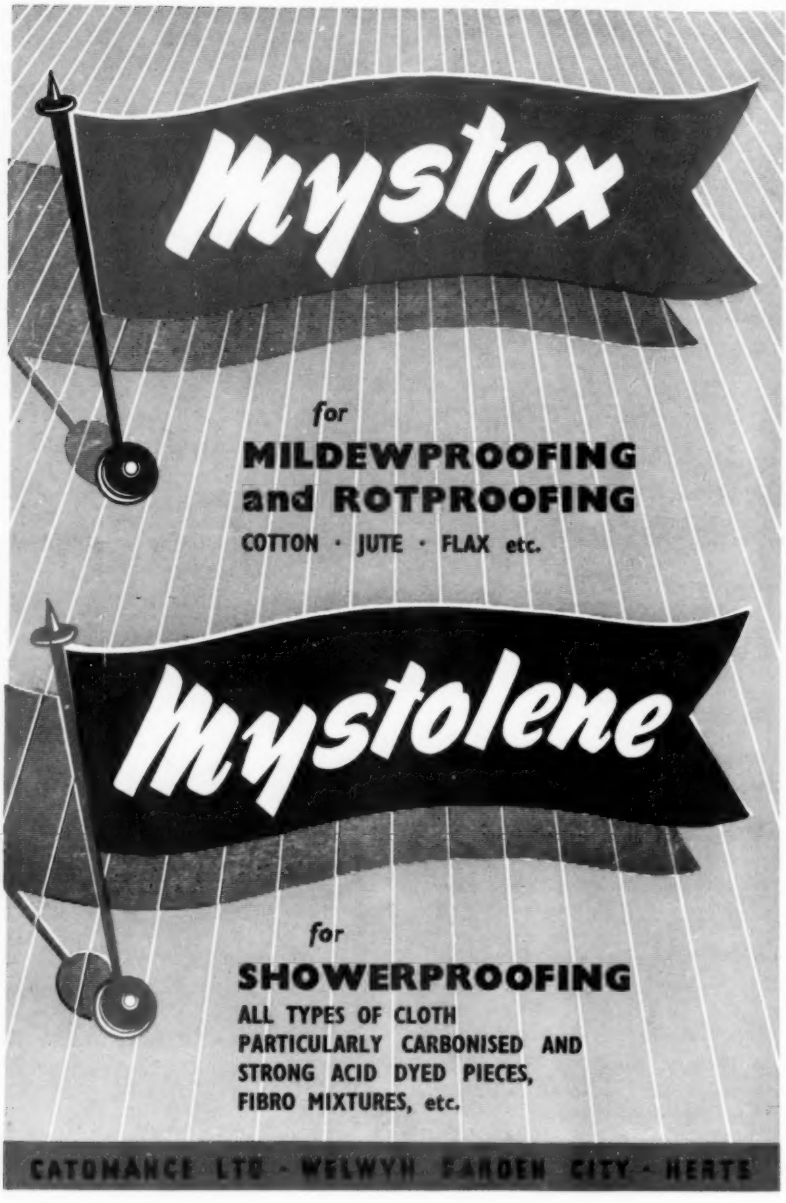
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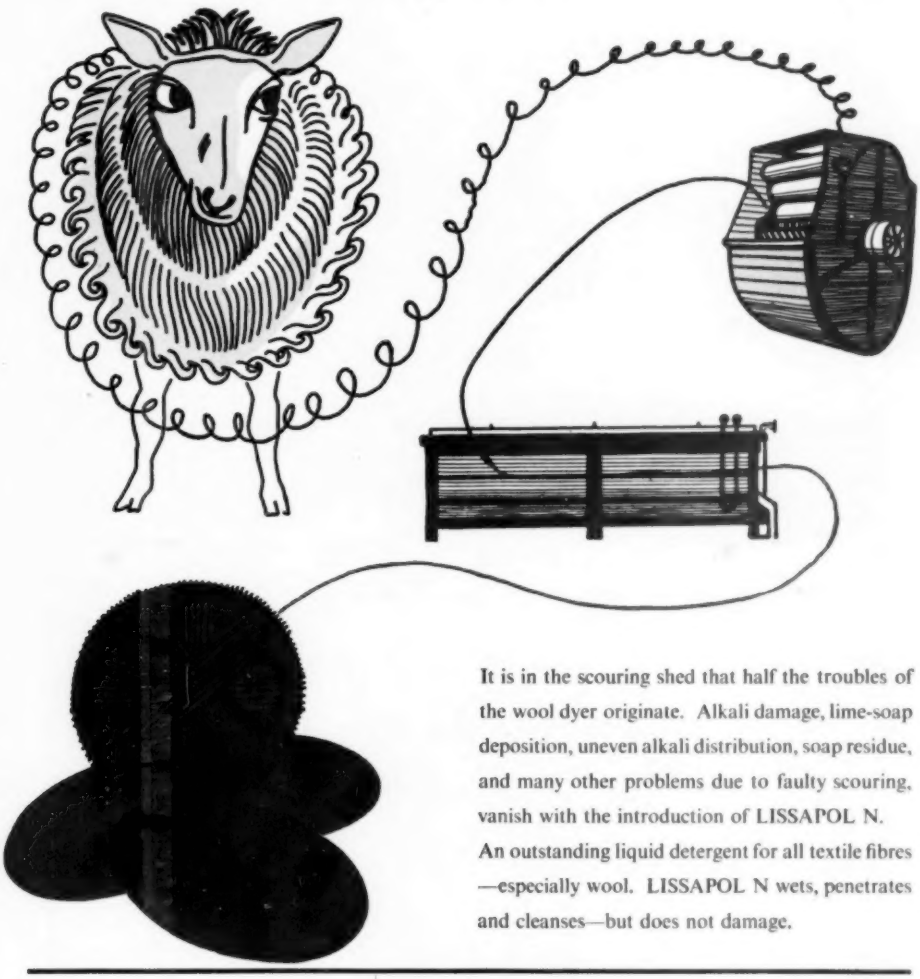

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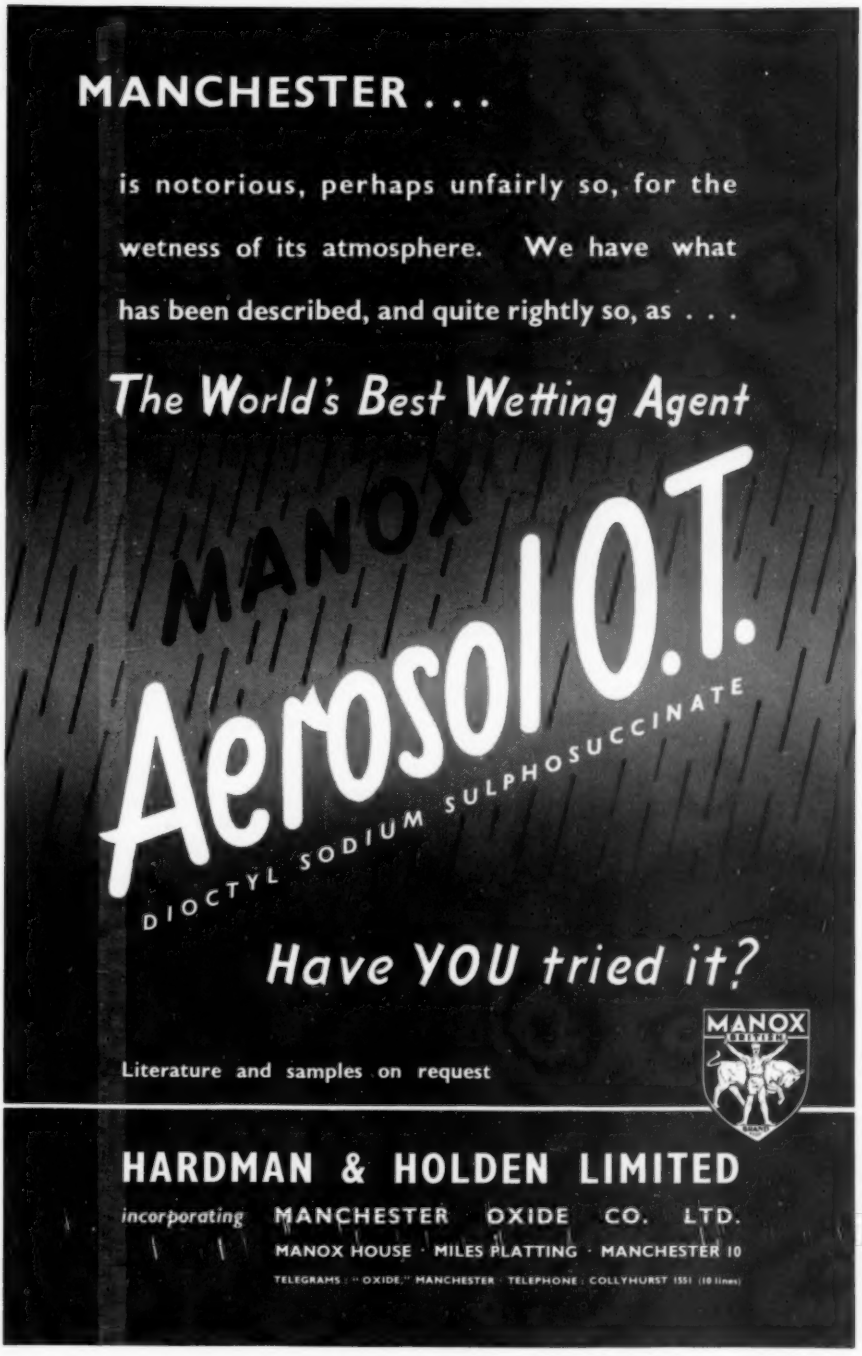
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Textile Chemistry in the Laboratory

By B. E. Hartsuch. Pp. x + 75. New York: John Wiley & Sons Inc. London: Chapman & Hall Ltd. 1950. Price, 13s. 0d.

This is a companion volume to the same author's *Introduction to Textile Chemistry* (above). As stated in the preface, "the purpose of this laboratory manual is not to make expert textile chemists of students, but to give the opportunity of carrying out some visual and manual experiments that will correlate the fundamental facts and concepts of textile fibres with their behaviour in practice and use". Detailed directions for carrying out some fifty-five experiments are given, and the average student will have no difficulty in obtaining satisfactory results. The experiments are classified under the following headings— the chemistry of cellulose, the chemistry of wool, laboratory examination of hard water, tests on soap, and work on silk. The remaining sections of the book are devoted to the identification of fibres and the preparation of solutions and reagents required for the course. Wherever possible reference is made to the appropriate page in the author's *Introduction to Textile Chemistry* to direct students to more detailed treatment of the theoretical background to the experiment.

The work is well set out, with blank spaces to be filled in by the student, and will serve as a sound basis for an elementary course in practical textile chemistry. The scheme could be operated in most textile departments with very little chemical equipment, and Professor Hartsuch has performed a useful service in preparing such a list of comparatively simple experiments. Many teachers and students will find the book most helpful.

C. S. Whewell

Artificial Fibres

By R. W. Moncrieff. Pp. x + 313. London: National Trade Press Ltd. 1950. Price, 25s. 0d.

This book may be recommended to dyeing technologists who wish to acquire a chemical background to the present range of rayon and synthetic fibres encountered in practice and also in theory (i.e. those fibres available in experimental but not bulk quantities). The book should also prove of value to general textile technologists with some knowledge of chemistry. Since the author is a chemist, it is understandable that his approach to the subject is chemical and physicochemical, and much of the contents of the book will not, therefore, be appreciated by the general textile technologist not possessing chemical knowledge. The book should be of value to chemical students who are desirous of entering the textile industry particularly concerned with rayon or synthetic fibre production.

In a future edition of the book the author would be strongly advised to omit all dyeing details, lists of dyes and dyeing machinery, and merely give references to authorities on the subject to which the reader could refer. These remarks apply particularly to the section devoted to the dyeing of viscose rayon. In this section a summary is given of some of the early, but now outdated, tests used for classifying direct dyes, but no reference is made to the more recent Society of Dyers and Colourists' classification of direct dyes. Although the earlier levelling tests referred to were those devised by C. M. Whittaker and his collaborators, whose more recent work (not quoted by the author) paved the way for the S.D.C. classification, there is no reference throughout any of the dyeing sections or in the bibliography to this pioneer in rayon-dyeing technique. A reference to the most recent edition of *Dyeing with Coal Tar Dyestuffs* by C. M. Whittaker and C. C. Wilcock, particularly to the section on the dyeing of rayons, would satisfactorily replace pp. 118–123 of Mr. Moncrieff's book. A large proportion of this section is devoted to basic dyes, which are very rarely used for dyeing viscose rayon, whereas the vat dyes, an extremely important class for dyeing this fibre, receive scant mention in a small paragraph.

The author has attempted to cover much too wide a field and has relied too much upon his own knowledge. This approach has an advantage from many points of view, as, for example, in Part IV of the book, devoted to "Synthetic Fibres", one immediately becomes aware of the author's considerable insight into this subject from his personal experience and investigations. There is no attempted re-hash of the literature and his summing up of the potentialities of the various synthetic fibres has the ring of authenticity and conviction. In some of the other sections, however, the bibliography is weak, e.g. that dealing with the structure of cellulose. There is nowhere a reference to the work of W. N. Haworth and his collaborators, nor is there any reference to the classical X-ray investigations of Sponsler and Dore and of Meyer and Mark, although some of their experimental results are used in the text.

The book is divided into four parts— Part I, dealing with the structure and properties of fibres, contains some excellent chapters, particularly chapters dealing with orientation and crystallinity, and the influence of orientation and chemical constitution on fibre properties.

Part II deals with the regenerated cellulosic and alginic fibres. The historical approach to this section is excellent, as also is the chemical and production background. The chapter on alginate fibres is particularly commendable.

Part III is devoted to the regenerated protein fibres. This section gives a satisfactory account of the present position, but does not sufficiently stress the possibilities of these fibres in blend with viscose rayon staple. Mention of a probable selling price for a fibre not yet in commercial production is made and is apt to be misleading during these times of rapid cost increases.

Nothing but praise is due for Part IV, "Synthetic Fibres", which is succinct. It contains an interesting chapter on glass fibres, which, however, omits to mention the "coronising" process, a special finish developed in America to give a soft handle and dimensional stability.

Part V, "Processing", is a somewhat sketchy section containing information on production, crease-resist finishing, and an unsatisfactory

chapter on dyeing. The last chapter, concerned with the identification and estimation of fibres, is a very helpful summary.

The chapters on synthetic polymer fibres and on chemical constitution, orientation, and fibre properties of rayon and synthetic fibres more than justify the price of the book. H. A. THOMAS

Kunstseide und Stapelfaser

By A. Zart. Pp. vii + 177. Darmstadt: Verlag von Dr. Dietrich Steinkopff 1950. Price, paper, DM. 17.50; cloth, DM. 19.50.

Dr. Zart's book may be recommended to students of chemistry and physics who wish to enter the rayon production industry. The book is concerned mainly with the production and chemical and physical properties of the cellulosic rayons, a field in which the author has had considerable practical experience from the early days of production of cuprammonium and viscose rayon up to the present time. He has included a very short chapter on the regenerated protein fibres and a more convincing chapter on the synthetic polymer fibres. The text is well illustrated with structural formulæ, graphs, a few photomicrographs, and some useful flow diagrams and machine drawings of rayon production machinery and testing equipment. The bibliography is reasonably complete, but there is an outstanding omission, since there is no reference to the work of W. N. Haworth and his collaborators in the carbohydrate and cellulose field, although the author utilises some of Haworth's terminology and structural representations.

The book is written in German, and the reading of it should, therefore, prove a useful exercise for science students. H. A. THOMAS

Die neuzeitlichen Textilveredlungs-Verfahren der Kunstfasern

1. Lieferung

By F. Weber and A. Martina. Pp. viii + 124. Vienna: Springer-Verlag 1950. Price, 31s. 6d.

The authors of this work have set themselves the task of making a complete review of the patent, technical, and scientific literature dealing with and related to textile finishing over the period from 1939 to 1949–50. It is proposed to issue the whole volume in about six parts, the last being promised by the end of this year; the authors state that they expect it to extend to about 700 pages, reviewing 7500 patents and 1000 original literature references. The anticipated price is about £10 or $24.

The first section, reviewed here, deals with the production and physical and chemical properties of the rayons and certain modified natural fibres. The authors suggest in their preface that such a foundation is an essential ingredient of an intelligent approach to the chemistry and technology of textile finishing. After reading this section, the reviewer feels that this point has been satisfactorily made, since all with an interest in textile chemistry or technology will find here much that is new and valuable. The fibres are treated one by one; some indication of the scope of the work may be realised in noting that about 1500 patents and 300 literature references are cited.

Future parts promised will cover bleaching, dyeing, printing, finishing, scouring and other operations.

From what has been said it is apparent that the first section will not have the interest of the succeeding sections for the dyer and colourist, but it may indeed be used to assess the probable value of the work as a whole. A summarising compilation of this type can be judged only in respect of accuracy and completeness; in those fields where the reviewer has first-hand knowledge the authors appear to have omitted little of importance; and the accuracy is at least up to the standards achieved in books of this type.

If the levels of achievement reached in this section are maintained in the more purely technical sections, the complete volume is likely to take its place in all textile libraries. The topics promised treatment in the detailed prospectus offer a wide field of interest to the textile chemist, so that an acceptable guide to the technical literature of the war decade will be welcome.

The printing is good, and there is an almost complete absence of printer's errors; the arrangement is clean, and the text should present no difficulties to the average English reader of technical German. The price, though high, is not out of scale of similar books.

This work is recommended to all interested in Textile Chemistry as a source-book covering material not otherwise collected into one place.

T. H. MORTON

Aminoplastics

By C. P. Vale. Pp. 250. London: Cleaver-Hume Press Ltd. 1950. Price, 12s. 6d.

For its size, this book gives a very good general review of the subject. Each chapter is concluded with an extensive list of references, and at the end of the book there is a very useful bibliography.

The first three chapters, dealing with the chemistry of amine–aldehyde resin formation, contain useful information on commercial methods of producing the raw materials for this group of plastics, viz. urea, formaldehyde, melamine, thiourea, etc. Excellent summaries of the latest theories of resinification for urea–, melamine–, and thiourea–formaldehyde condensation products are also included. In the chapter dealing with miscellaneous amino resins, one might have expected to find some mention of the aniline–formaldehyde resins, especially since the guanidine–formaldehyde resins are considered important enough to be included, but in the introduction to the book the author gives his reasons for excluding them.

The author states that "details of the manufacture of moulding powders relate generally to closely guarded processes". This is understandable, but it is a factor which renders it difficult to write on this subject and at the same time avoid presenting the reader with a series of extracts from the patent literature in such a manner that the result is, to say the least, somewhat confusing. In the chapter dealing with this subject the author maintains a good balance between patent abstracts and his own statements, and is to be congratulated on

the lucidity of the result. A useful section on the properties of moulding powders is included.

The chapters on moulding processes and physical properties of moulded materials are presented in a comprehensive manner considering their limited size. There might have been some mention of the Society of Dyers and Colourists' method of measuring light fastness, set out in *B.S.*1006, Parts 1 and 2, which allows a definite rating to be given to the light fastness of plastics materials.

The chapters dealing with lacquers and adhesives give an excellent account of the chemistry of the processes involved; but from the technical point of view the treatment is somewhat brief in comparison with the amount of space allotted to moulding resins. More could have been said, for example, on the formulation and testing of lacquers, and it is a pity that no mention is made of the important German war-time development of unmodified adipic alkyds, which combine the functions of modifier and plasticiser.

J. K. AIKEN
A. HILL
R. MATHER

Reports on German and Japanese Industry

Anorgana G.m.b.H., Gendorf
Aliphatic Sulpharylides, Polyacetals, Alkylated Empyreumatic Oils, Alkylphenoxyacetic Acids, *NN'*-Ethylenecureas, Polyethylene Glycol Ethers, *N*-Substituted Aspartic Acids—Textile Auxiliary Products

FD 1209/50* (PB 70,344).

Microfilm of 13 papers written in German in 1936–44.

Softening, Emulsifying, and Other Textile Auxiliary Agents

FD 1172/50* (PB 70,344).

Microfilm of 11 papers written in German in 1936–44. Among them are—

(1) *Softening Agents based on Iminazolines—II*
(2) *NN'-Ethyleneureas—Properties and Uses as Textile Auxiliary Agents*

Polyacrylhydroxyalkylamides and Monomeric Ethyleneimine Compounds as Auxiliary Agents for Textiles and Leather, Intramin, Trilon A

FD 1208/50* (PB 70,344).

Microfilm of 7 papers written in German in 1936–43 and including—

(1) *Use of Polyacrylhydroxyalkylamides in Sizing Rayon, Pigment Fixation, Screen Printing, etc.*
(2) *Monomeric Ethyleneimine Compounds in Textile Finishing*
(3) *Contribution to the Knowledge of the Calcium-sequestering Property of Trilon A and Ma* 416.

Continuous Hydroxyethylation, Amines from Olefins, Urethans, Chloroalkylpolyglycol Ethers, Aliphatic Amines, Acrylic Acid Derivatives—Textile Auxiliary Agents

FD 1207/50* (PB 70,344).

Microfilm of 10 papers written in German in 1936–43.

Igepons, Medialans, Leonil S—Wetting Agents

FD 1168/50* (PB 70,344).

Microfilm of 11 papers written in German in 1936–41.

Washing Agents

FD 1169/50* (PB 70,344).

Microfilm of 21 papers written in German in 1934–37. They deal with the physical chemistry of soap solutions and various problems connected with the manufacture and use of synthetic detergents, including the incorporation of Tylose and Trilon A into such products.

Finishing of Rayon Fabrics—Dyes and Pigments

FD 1170/50* (PB 70,344).

Microfilm of 11 papers written in German during 1936–43. They include—

(1) *Use of Formaldehyde Condensates from Diamines, Diureas, and Diurethan in Rayon Finishing*
(2) *Finishing of Spun Rayon with Formaldehyde–Low-molecular Amide Condensates*
(3) *Catalysts and Drying Conditions in Formalising Processes*
(4) *Sodium Cellulose Glycollate and Hydroxyethanesulphonate (Tylose HB and SG) as Hardness-sequestering Agents*
(5) *Bifunctional Products in the Processing of Textiles.*

C. O. C.

Anorgana G.m.b.H., Gendorf
Dyes and Intermediates—Identification of the Components of Dyes—Synthetic Tannins

FDX 893* (PB 84,192; Microfilm B 1).

A large number of reports written in 1911–1921, mostly dealing with the manufacture of intermediates and dyes and synthetic tanning agents, checking of patents applied for by competitors, analysis of competitors' products, and investigation of the constitutions of a number of dyes.

C. O. C.

German Practice in making Acetate Rayon

FD 62/51* (PB 1352).

Short report written in 1945 by an Allied investigator. It deals with the manufacture of cellulose acetate flake, acetic anhydride, and filament and staple acetate rayon at the I.G.'s works at Dormagen, Rhodiaseta A.-G. at Freiburg, and Dr. Alexander Wacker G.m.b.H. at Burghausen.

C. O. C.

I.G. Farbenindustrie A.-G., Leverkusen and Höchst
Fibre-forming Polymers and Intermediates

FD 63/51* (PB 1354).

Précis of information obtained in 1945 by Allied investigators on the manufacture of PeCe fibre and the various Perlons.

C. O. C.

Japanese Technique in Artificial Fibres and Allied Fields

FD 15/51* (PB 100,358).

Microfilm copy (118 frames) of a report written in 1949 by an Allied investigator. On the whole, the Japanese rayon industry was behind that of other countries. It had, however, developed a high-tenacity low-swelling staple fibre. Vinylon (Kuravillon), a formalised polyvinyl alcohol, is better than Vinyon for textile requirements. A polyamide fibre, Amilan, resembles nylon. No developments of interest were noted in the pulp and paper industries. Progress had been made in the employment of ramie in textiles.

C. O. C.

* Material available for inspection at, and photocopies available from, T.I.D.U., Board of Trade, Lacon House, Theobalds Road, London W.C.1, or (under PB No.) from Office of Technical Services, Department of Commerce, Washington D.C., U.S.A.

Abstracts from British and Foreign Journals and Patents

(The Titles of Patents are abridged and modified)

I— PLANT; MACHINERY; BUILDINGS

Chemical Engineering Unit Operations Review. *Ind. Eng. Chem.*, **43**, 37–116 (Jan. 1951).

Operations reviewed are absorption and humidification, adsorption, centrifugation, crystallisation, high-temperature distillation, high-vacuum distillation, drying, evaporation, solvent extraction, filtration, fluid dynamics, heat transfer, ion exchange, materials handling, mixing, and size reduction. C. J. W. H.

[Particle] Size Reduction— Jet Mills, Homogenisers, Particle Size Measurement, Explosion and Fire Risks. R. V. Riley. *International Chem. Eng.*, **32**, 22–26 (Jan. 1951).

A review with 70 references. C. O. C.

PATENTS

Spinning Gun for Production of Filaments and Method of making Non-woven Fabrics. F. W. Manning. *U.S.P.* 2,522,526, *U.S.P.* 2,522,527.

Thread-advancing Reels. British Celanese Ltd. *B.P.* 648,180.

Applying Liquids uniformly to Running Yarns. Celanese Corpn. of America, F. F. Sutherland, and D. K. Wilson. *U.S.P.* 2,521,807.

Mounting for Yarn Packages in Yarn Driers, Dyeing Machines, etc. Smith, Drum & Co. and H. F. Liedtke. *U.S.P.* 2,524,629.

Drying of Yarn. A. Ashworth. *B.P.* 648,971.

Rapid and cheap drying is obtained by hanging the hanks on rods across a drying oven which is totally enclosed except for controllable air inlets at the bottom. The yarn is so tightly packed across the oven as to divide it into two parts, the lower part containing a heater for the yarn and the air inlets, the upper part being connected to an extraction fan, which maintains a partial vacuum above the yarn. C. O. C.

Use of Ultrasonic Vibrations in Washing Textiles. S. B. Wiczer. *B.P.* 648,609.

High-intensity ultrasonic waves of frequency $> 16{,}000$ cycles/sec. are passed into the washing liquor for 1–20 min,, the liquor being preferably continuously changed during the treatment. C. O. C.

Hydraulic Drives for Web Unwinding and Winding Machines. Oilgear Co. *B.P.*648,269.

Rollers for Printing and Textile Machinery. Dayton Rubber Co. *B.P.* 648,275.

A roller having a high suction effect and great resistance to wear consists of a layer of sponge rubber surrounding an inner layer of hard rubber. C. O. C.

Heating of Calender Rolls. American Viscose Corpn. *B.P.* 647,102.

The roll is made of a heat-conducting material and has one closed end. Inside it there is a stationary heating device which does not touch the walls of the roll. A flowable heat-conducting material in the roll covers at least some of the heating device. The other end of the roll has a central opening through which there protrudes into the roll an outwardly flared member, which surrounds this central opening so as to deflect the heat-conducting material away from the opening. This prevents leakage of the heat-conducting material at the opening. C. O. C.

Drying. G. Ilune. *B.P.* 648,388.

The difference between the vapour pressure of the fluid to be eliminated in the material to be dried and the vapour pressure of that which has passed over into the atmosphere of the drying chamber is varied while all other drying factors, e.g. temperature of the drying air, flow of the air through the chamber, and primarily the quantity of fluid to be eliminated from the material per unit of time, are kept constant. This renders drying easily controllable, so that the material can be dried almost at the limit permissible with a small margin of safety, and the time required is thus reduced to a minimum. C. O. C.

Tenter. F. B. Morrill. *B.P.* 648,465.

A new arrangement and combination of parts make it economically advantageous to extend horizontal pass tenters lengthwise. This enables such tenters to be rebuilt or built over to attain at the same relative working cost a rate of production previously impossible. The cost of doing this is far less than that of using separate tenters having the same output. C. O. C.

Cloth-shearing Machines. Zbrojovka Brno N.P. *B.P.* 647,589.

A displaceable pile-raising brush is mounted on pivotable levers in front of the knives, so that it is readily raised by the operator when a seam or the like comes along. The displacement of the levers carrying the brush is limited in the direction of the leading edge of a cloth tension rail by means of an adjustable screw. C. O. C.

Screen Printing Machines. L. Martin. *B.P.* 647,097.

The material to be printed is carried on an endless belt which travels over a flat supporting surface on the machine. Continuously driven reciprocating mechanism including an adjustable throw crank operating through a one-way clutch upon the belt drive causes the belt to travel through steps of predetermined length. A printing screen frame supported over the bed is automatically moved into and out of contact with the material to be printed during the intervals when the belt is stationary. C. O. C.

Nylon-preboarding Machine. Paramount Textile Machinery Co. *B.P.* 646,826.

The machine is constructed according to *B.P.* 602,262 and 615,123 (cf. J.S.D.C., **64**, 348 (1948); **65**, 367 (1949)), several form carriages mounted on a main carriage moving on parallel tracks being introduced alternately into a horizontal retort. The retort door and the steam delivery and exhaustion are operated by an electrical control. A safety device is incorporated in the movement of the door, and means are provided for locking the main carriage while the operative is working with the forms. G. E. K.

Conveyor-type Washing or Dry Cleaning Machine. American Laundry Machinery Co. and G. W. Johnson. *U.S.P.* 2,523,304.

The machine comprises coaxial, circular end heads spaced apart along a horizontal axis and an endless belt having an upper operating stretch and a lower idle stretch. The belt is supported so that opposite marginal end portions of one face of its operating stretch engage the lower portions of the end heads so as to form leak-proof seals. An end loop of the belt normally extends vertically upwards for the cleansing process. This belt loop can be adjusted to incline upwards and away from the axis, so that it can act as a work-discharge conveyor. A cylindrical wall connects the end heads so as to form an overall drum, there being a charging opening in this wall. The belt can as a whole be lifted up above its operating stretch to enable the work to be placed on it through the charging opening. *U.S.P.* 2,523,305.

A drum having an opening in its cylindrical wall is mounted to swing about its horizontal axis. Inside the drum is an endless belt having two end loops spaced apart to provide a gap opposite the opening and outer and inner stretches, the latter mounted upon and moving with the peripheral portions of the end heads. The end loops and outer stretch of the belt are supported upon a series of parallel shafts. The belt acts as both an agitator and a conveyor for the work. C. O. C.

Stretching and Drying Device for Garments. G. W. Johnson. *B.P.* 648,841.

Garment Steaming and Drying Apparatus. J. C. Shaw. *B.P.* 646,889.

Coats, overcoats, etc. may be steamed or dried by mounting them over a hollow, vertical, rotatable porous form, which may be tilted to facilitate operations. An inner chamber is subdivided to permit controlled flow of air or steam through any desired section of the garment. Drying is effected by either suction or a flow of heated air. G. E. K.

Garment-pressing Machines. Prosperity Co. Inc. *B.P.* 647,600.

Alphabetical List of New Products developed since November 1949 [in U.S.A.]. (IV, p. 157.)

II—WATER AND EFFLUENTS

PATENTS

Compositions for inhibiting Foaming in Boilers. National Aluminate Corpn. *B.P.* 648,099.

High-mol.wt. monoethers of polyoxyalkylene glycols are very effective anti-foaming and anti-fuming agents for steam generators. C. O. C.

Breaking Emulsions to recover Wool Grease. R. Shawcross. *U.S.P.* 2,522,140.

Effluents from wool scouring are passed through a bed of an insoluble, infusible, acid, sulphonated phenolic resin. This causes the wool grease to be deposited on the resin, whence it is removed by periodical washing with a solvent. C. O. C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Preparation of Sodium Carboxymethyl Cellulose. R. R. McLaughlin and J. H. E. Herbst. *Canadian J. Res.*, **28 B**, 731–736 (Nov. 1950).

In an investigation of the preparation of sodium carboxymethyl cellulose from cellulose, sodium hydroxide, and sodium chloroacetate in the presence of water, the effects of temp., excess of sodium hydroxide, ratio of water to cellulose, time of mixing, and manner of addition of the reagents were studied. The highest degree of substitution attained was 2·8. W. K. R.

Determination of the Degree of Substitution of Carboxymethyl Cellulose over the Entire Substitution Range. R. R. McLaughlin and J. H. E. Herbst. *Canadian J. Res.*, **28 B**, 737–744 (Nov. 1950).

Published methods for the determination of the degree of substitution of carboxymethyl cellulose were found to be unsatisfactory for samples of a high degree of substitution. Two methods were developed which gave satisfactory results over the entire substitution range— (*a*) impurities are removed from crude sodium carboxymethyl cellulose by dialysis or washing with ethanol, and the sodium content is determined; (*b*) cupric carboxymethyl cellulose is precipitated under controlled conditions and purified, and the copper content is determined. The degree of substitution is calculated from the metal content. W. K. R.

Preservation of Sodium Carboxymethyl Cellulose from Microbiological Degradation. S. O. Pehrson. *Svensk Papperstidning*, **53**, 726–732 (30th Nov. 1950).

The viscosity of heat-sterilised solutions of sodium carboxymethyl cellulose stored for long periods under aseptic conditions does not normally decrease, but unsterilised solutions often show a rapid loss of viscosity and a variety of undesirable changes, including discoloration, deposits, and disagreeable odours, due to microbiological action. Experiments have shown that 1–10 mg. of phenylmercuric acetate (100%) or 100–200 mg. of Na pentachlorophenate per litre can safely be employed, being added to the water used for the preparation. In some instances 100 mg. of the Na salt of dihydroxydichlorodiphenylmethane per litre is recommended. S. V. S.

Organic Derivatives of Titanium. I—Preparation of some Ortho-esters. N. M. Cullinane, S. J. Chard, G. F. Price, and B. B. Millward. *J.S.C.I.*, **69**, S 38–S 40 (Supplementary Issue No. 1, 1950).

The preparation of alkyl orthotitanates by the action of titanium tetrachloride on an alcohol in the presence of sodium or ammonia is unsuccessful in the case of alcohols, such as *tert.*-butanol, which react readily with hydrogen chloride. Titanium esters of these alcohols, however, may be prepared, in good yield, by using a mixture of alcohol and benzene, with pyridine to absorb the HCl, and then treating with ammonia W. K. R.

Effectiveness of some Chlorinated Hydrocarbon Insecticides against the Leather Beetle *Dermestes maculatus* Deg. J. N. Turner. *Bull. Entomol. Research*, **41**, 289–299 (1950): *Chem. Abs.*, **45**, 300 (10th Jan. 1951).

γ-Benzene hexachloride and chlordan are much more toxic to this insect than is DDT, which has comparatively little effect on either eggs, larvæ, or adults. C. O. C.

PATENTS

High-viscosity Starch Ethers. General Mills Inc. and O. A. Moe. *U.S.P.* 2,523,709.

Carboxyalkyl starch ethers exhibiting high viscosities in dilute aqueous solution are obtained by elimination of water-soluble electrolytes and adjustment of the pH to below that of the alkali-metal carboxyalkyl starch derivative all of whose carboxyl groups are present as alkali-metal salts. The high viscosity of such solutions can be destroyed by addition of water-soluble electrolytes, but is unaffected by addition of water-insoluble salts, polyhydric alcohols, or sugars. C. O. C.

Chemically Modified Starches. United States Rubber Co., D. L. Schoene, and V. S. Chambers. *U.S.P.* 2,524,400.

Starch and dextrin react with vinyl sulphone to yield products which are insoluble in boiling water. The reaction is carried out preferably in aqueous solution in presence of a basic catalyst. The reaction may be carried out *in situ* on a fabric sized with starch, when a stiff finish is produced which resists washing. C. O. C.

Stable Sodium Alkyl Sulphate Solutions. N. V. De Bataafsche Petroleum Maatschappij. *B.P.* 646,827.

The tendency of sodium alkyl sulphate solutions to gel or crystallise, particularly at low temperatures, may be reduced by adding potassium ions, preferably as a basic compound when the sulphation product is neutralised. The molecular ratio of potassium and sodium compounds is preferably about 1 : 3. G. E. K.

Detergent able to sequester Metal Ions. F. C. Bersworth. *U.S.P.* 2,524,218.

Compounds of formula—

$$A\cdot ND\cdot[CH_2]_n\cdot[ND\cdot[CH_2]_n]_m\cdot ND\cdot A$$

(n = 2 or 3; m = 0 or a whole number; D = $-CH_2\cdot COOH$ or $-CH_2\cdot CH_2\cdot COOH$ or an alkali-metal salt thereof; A = a hydrocarbon radical containing > 1 C and < 3 H atoms replaced by $-CH_2\cdot CH_2\cdot COOR^1$, $-CH_2\cdot CH_2\cdot COOM$, $-SO_3H$, $-SO_3M$, $-O\cdot R^1$, or $-O\cdot R^3\cdot O\cdot R^1$ (R^1 = H or Alk, R^3 = alkylene, M = alkali metal), such groups being spaced at least 2 C atoms from the N atoms), e.g.—

$$\begin{array}{ll} NaOOC\cdot CH_2 & CH_2\cdot COONa \\ & | \\ NaO_3S\cdot CH_2\cdot CH_2\cdot N\cdot CH_2\cdot CH_2\cdot N\cdot CH_2\cdot CH_2\cdot SO_3Na & \end{array}$$

are excellent as detergents and for sequestering metal ions and are completely miscible with soap. C. O. C.

Germicidal Deionising Agents for Laundering Clothes. F. C. Bersworth. *U.S.P.* 2,524,219.

The mono- to tetra-quaternary ammonium salts of ethylenediaminetetra-acetic acid are strong germicides soluble in alkaline soap solutions. They act as sequestering agents for metal ions, and are especially suitable for use in rinsing dirty soap solution from washed goods, a much whiter wash being obtained. C. O. C.

Concentrated Detergent Compositions. N. V. De Bataafsche Petroleum Maatschappij. *B.P.* 646,948.

A concentrate of secondary alkyl sulphates containing < 6 C is obtained by salting out in presence of an organic solvent (b.p. > 100°c.) containing one or more polar groups, e.g. *cyclo*hexanol. An upper layer comprising the concentrated ester salt and solvent separates, from which the latter is removed by distillation. G. E. K.

Detergent Composition. Scottish Oils Ltd. and D. Stewart. *B.P.* 646,765.

Metal alkyl sulphates of low water solubility are produced by sulphation of a distillation fraction of shale oil (b.p. 270–360°c.) with 10–20% by vol. of sulphuric acid (96–100%) at < 20°c. followed by neutralisation with sodium carbonate. Incorporating a filler, e.g. kieselguhr, a binder, e.g. bentonite, and a refined mineral oil yields compositions suitable for removing mineral lubricating oils and sludges from operatives' hands. G. E. K.

Textile Lubricating and Conditioning Agents. British Celanese Ltd. *B.P.* 649,148.

A composition for lubricating and conditioning textile fibres, particularly organic esters of cellulose, comprises a mineral oil, a long-chain aliphatic alcohol, e.g. lauryl alcohol, an alkylphenol, e.g. diamylphenol, an alkylolamine, e.g. triethanolamine, a neutralised sulphonated long-chain aliphatic acid, a neutralised sulphonated vegetable oil, an alkylolamine ester of a long-chain aliphatic acid, and a neutralised partial ester of phosphoric acid with a long-chain aliphatic alcohol. J. W. B.

Capillary-active Substances and Softening Agents. Sandoz Ltd. *B.P.* 648,768.

Compounds of formula $R^1{\cdot}CO{\cdot}NH{\cdot}R^2{\cdot}NR^3{\cdot}R^4{\cdot}SO_3Y$ (R^1 = Alk of > 9 C; R^2 = alkylene or hydroxyalkylene of 2–6 C; R^3 = Alk of 1–6 C or hydroxyalkyl of 2–6 C; R^4 = alkylene or hydroxyalkylene of 2–6 C in which the C chain may be interrupted by an O atom; Y = a cation), e.g.—

$$C_{17}H_{33}{\cdot}CO{\cdot}NH{\cdot}C_2H_4{\cdot}N(C_2H_4OH){\cdot}CH_2{\cdot}CH_2{\cdot}SO_3Na$$

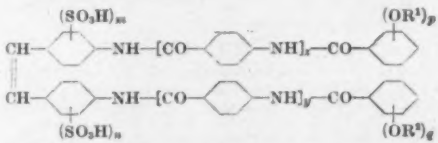
are good wetting, dispersing, penetrating, etc. agents in neutral, acid, or alkaline solutions and are also suitable as softening agents. C. O. C.

Colloidal Silicate Emulsifying Composition. R. T. Vanderbilt Co. Inc., W. K. Feustel, and F. E. Hutchins. *U.S.P.* 2,523,204.

A mixture of a high-gel magnesium bentonite (6–15 parts by weight), a high-gel aluminium bentonite (20–60), and a medium-gel aluminium bentonite (30–70) has high suspending or emulsifying capacity and good rate of gelation, and the gels, suspensions, or emulsions formed by its use are little affected by changes in temperature. C. O. C.

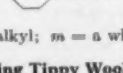
Blue-fluorescent Effects and Agents therefor. Lever Brothers & Unilever Ltd., L. N. Savidge, and R. Thomas. *B.P.* 647,718.

Compounds of formula—



(R^1 and R^2 = Alk, Ar, or aralkyl; x and y = 0 or a small number; n, m, p, and q = whole numbers) are substantive to cellulose and give good optical bleaches. *B.P.* 647,759.

The above compounds are made by treating appropriate stilbene derivatives with acyl chlorides of formula—



(R = Alk, Ar, or aralkyl; m = a whole number). C. O. C.

Assistants for Dyeing Tippy Wool. Arkansas Co. Inc., A. A. Cook, and I. Sapers. *U.S.P.* 2,524,041.

An agent which assists in the level dyeing of tippy wool consists of a synergistic combination produced by heating 20–50 parts by weight of a polyethylene glycol aliphatic ester of mol. wt. 400–6000 and containing 1–2 mol. of acid per mol. of glycol with 5–20 parts of a condensate of 1 mol. of a water-soluble C_{2-18} aliphatic amine and 1–2 mol. of a C_{10-22} aliphatic acid. The condensate is produced by heating the components together at 180–220°c. for 1–4 hr. until there is not $> 1\%$ free fatty acid present. The combination should have pH 6–7. C. O. C.

Guanidine Thiocyanate as Combined Flame-resisting and Plasticising Agent for use with Hydrophilic Sheet Materials. American Viscose Corpn. *B.P.* 647,532.

Guanidine thiocyanate exerts both a softening and a fire-retarding action when incorporated into sheets of regenerated cellulose, cellulose esters or ethers, casein, leather, gelatin, hydrophilic synthetic resins, etc. C. O. C.

Water-dispersible Urea–Formaldehyde Resins especially suitable for treating Cellulosic Textiles. Beck, Koller & Co. (England) Ltd. and P. S. Hewett. *B.P.* 648,354.

Urea (1 mol.) is heated with formalin (1·9–2·1 mol. HCHO) containing acid impurities, the pH being controlled by simultaneous use of sodium acetate and ammonia, so that the initially alkaline dispersion gradually becomes acid as the ammonia is driven off until pH 5 is reached. The product has particularly good water-resistance, and while capable of rapid and thorough conversion is stable enough to use in textile finishing. C. O. C.

Halogenated Aromatic Amine–Formaldehyde–Dicyandiamide Resins— Mildew- and Moth-proofing, Dispersing, and Anti-gas-fading Agents. General Aniline & Film Corpn. and R. T. Olsen. *B.P.* 647,220.

Formaldehyde (1 mol.) is treated with a primary or secondary aromatic amine containing at least one halogen atom (1 mol.), and the product then treated with dicyandiamide (1 mol. for each mol. of formaldehyde or amine used). The products are useful as mildew- and moth-proofing and dispersing agents and as inhibitors for gas fading. C. O. C.

Thermosetting Polyaryldiguanide–Formaldehyde Resins— Crease-resistant Agents. General Aniline & Film Corpn., A. L. Fox, H. L. Sanders, and R. T. Olsen. *B.P.* 647,222.

A resin formed by the process of *B.P.* 647,220 (above) is treated with at least 1 mol. of formaldehyde for each mol. of amine or dicyandiamide used in making it. The products are converted by heat into hard, infusible, water-insoluble, glass-like resins, but before being heated they are water-soluble and may be used to render textiles crease- and shrink-resistant. These halogenated products are much more stable to light than the corresponding unhalogenated products. C. O. C.

Silicyl Esters as Water-repellent Compositions. Silicon (Organic) Development Ltd., D. A. W. Hill, W. E. Langrish-Smith, and C Shaw. *B.P.* 647,923.

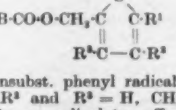
Application of a mixture of a silicon ester, e.g. methyl orthosilicate, and a base, e.g. monoethanolamine, either alone or in a suitable solvent, followed by stoving at 100°c. for a few min., renders textile fibres, paper, glass, ceramics, and metals permanently water-repellent. C. O. C.

Water-repellent Fabrics and Compositions. Montclair Research Corpn., Ellis-Foster Co., J. B. Rust, and W. B. Canfield. *U.S.P.* 2,524,022.

A wax and an aliphatic C_{16}–C_{22} primary amine are dispersed in an aqueous solution of a salt of a volatile base and a linear polymeric polycarboxylic acid. There are 0·15–0·95 mol. of amine per carboxyl group and 1–4 parts of wax per 1 part of polymeric acid and amine combined. Fabrics impregnated with the composition and then heated are given a permanent water-repellent finish of soft handle. C. O. C.

Rendering Textiles Insect-repellent. Monsanto Chemical Co., L. P. Kyrides, and F. B. Zienty. *U.S.P.* 2,519,383.

Compounds of formula—



(B = subst. or unsubst. phenyl radical; R^1 = H, Hal, CH_3, or C_2H_5; R^2 and R^3 = H, CH_3, or C_2H_5), e.g. 2-thienyl benzoate or salicylate, effectively condition textiles against infestation by mites, e.g. chiggers and lice, or insects. They are both more effective and faster to laundering than benzyl benzoate. C. O. C.

Pest- and Rot-proofing Agent. Nuodex Products Co. Inc. *B.P.* 648,400.

Alkyldimethylbenzylammonium naphthenates which are insoluble in water but soluble in benzene, e.g. *n*-dodecyldimethylbenzylammonium naphthenate, are very good rot-proofing agents, especially for cellulosic textiles. C. O. C.

Compositions having Fungicidal and/or Fungistatic Properties. Scientific Oil Compounding Co. Inc. *B.P.* 648,518.

A water-insoluble aluminium, alkaline-earth metal, or heavy-metal soap is heated with a heavy-metal or aluminium salt of a 5-, 6-, 7-, or 8-hydroxyquinoline. The product is readily dispersible in a number of liquids and so provides a convenient means for applying copper 8-quinolinoxide to textiles, leather, etc. or for incorporating it into coatings, paints, etc. C. O. C.

Mildewproofing Agents. Nuodex Products Co. Inc. *B.P.* 648,773.

The products obtained by treating a halogenated monohydroxybenzene having no other ring substituent with an aliphatic unsubst. primary amine having a C_4–C_{18} alkyl chain have excellent mildewproofing properties. They are soluble in oil but not in water. C. O. C.

Fungicidal Mercury Compounds. F. J. Sowa. *U.S.P.* 2,524,547.

Compounds of formula Ar·Hg·NH_2·R}X (R = Alk, *cyclo*alkyl, or aralkyl; X = anion) are excellent fungicides for treating textiles, adding to paints, etc. They are soluble in water and organic solvents. C. O. C.

Hot-melt Coating Composition. British Celanese Ltd. *B.P.* 646,945.

Cellulose propionate containing 0·1–0·5 free hydroxyl group per glucose unit is heated with 40–100% of a solvent plasticiser, e.g. dioctyl phthalate, or a mixture of a solvent plasticiser with a non-solvent plasticiser, to produce a homogeneous solution. The molten solution may be applied to metals, paper, fabrics, etc. by dipping, coating, or impregnating. E. C.

Vinylidene Chloride–Butadiene–Methyl Methacrylate Interpolymers. Dow Chemical Co., G. W. Stanton, and C. E. Lowry. *U.S.P.* 2,524,023

Vulcanisable products suitable as calendered coatings are obtained by polymerising a mixture of 40–70% vinylidene chloride, 15–30% diene hydrocarbon, and 8–35% methyl methacrylate. C. O. C.

Emulsions, Dispersions, and Solutions of Methacrylic Ester Copolymers. Vinyl Products Ltd., J. E. O. Mayne, and H. Warson. *B.P.* 648,001.

Methyl methacrylate is copolymerised with an alkali-metal methacrylate in aqueous medium, the molecular proportion of the latter ingredient being 8–98% of the mixture. The products have a wide range of uses, including the treatment of leather, paper, and textiles. C. O. C.

Plasticisers. Geigy Co. Ltd., H. Jones, and J. K. Aiken. *B.P.* 646,918.

Plasticisers suitable for use with cellulose acetate or triacetate and/or polyvinyl chloride comprise the cyclic acetals obtained from chloral and a 1:2-diol, e.g. 2-trichloromethyl-1:3-dioxolan trichloroethylidene glycerol, bis(tri-chloroethylideneglyceryl) adipate. E. C.

Driers. Nuodex Products Co. Inc. *B.P.* 648,638.

Hydroxy salts of drying metals, e.g. hydroxy metal naphthenates, can be dissolved in film-forming vehicles, e.g. linseed oil, by simple heating. C. O. C.

Alphabetical List of New Products developed since November 1949 [in U.S.A.]. (IV, below.)

Naphthopyridine Derivatives as Gas Fading Inhibitors. General Aniline & Film Corpn., H. W. Grimmel, and A. Guenther. (VIII, p. 166.)

IV— RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Alphabetical List of New Products developed since November 1949 [in U.S.A.]. *Amer. Dyestuff Rep.*, 39, 835–868 (11th Dec. 1950).

This covers dyes and pigments, textile chemicals, and equipment. C. J. W. H.

Studies in Light Absorption. IX— Relation between Absorption Intensities and Molecular Dimensions, and its Application to the Electronic Spectra of Polyenes and Polycyclic Benzenoid Hydrocarbons. E. A. Braude. *J.C.S.*, xiv (Dec. 1950).

Correction to previous paper, *J.C.S.*, 379–384 (Jan. 1950): *J.S.D.C.*, 66, 297 (May 1950).

Absorption of Light by Organic Colouring Matters. H. Kuhn. *Chimia* (Switzerland), 4, 203–218 (1950): *Chem. Abs.*, 45, 37 (10th Jan. 1951).

A review with 63 references. C. O. C.

Direct Iodination of Aromatic Compounds. D. H. Derbyshire and W. A. Waters. *J.C.S.*, 3694 (Dec. 1950).

The authors' recently described general method of direct bromination of aromatic compounds by bromine in the presence of a mineral acid and a soluble silver salt (*J.C.S.*, 573 (Feb. 1950)) is now extended to iodination. H. H. H.

Some Properties of the Diphenyl-4-diazocyanides and their 4′-Bromo Derivatives. H. C. Freeman and R. J. W. Le Fèvre. *J.C.S.*, 3128–3131 (Nov. 1950).

The rates of conversion of the respective labile into the stable forms of diphenyl-4- and 4′-bromodiphenyl-4-diazocyanides are measured at 25° and 45°C., and the energies of activation thus obtained agree with forecasts from previous work on the benzenediazocyanides, being 22·1$_5$ and 21·5$_4$ kg. cal. respectively. The dipole moments of the isomeric diphenyl-4-diazocyanides are found to be 4·5 and 5·5 D.; as with the two pairs of naphthalenediazocyanides (cf. *J.C.S.*, 333 (1949)), the stable form has the greater moment, compatible with a *cis–trans* relationship. Their ultra-violet absorption spectra in alcoholic solutions are similar but not identical, and show the differences expected for geometrical isomers. H. H. H.

Constitution of a Certain Nitration Product of 3:3′-Dichloroazoxybenzene. K. F. Keirstead and R. Gaudry. *Canadian J. Res.*, 28 B, 750–751 (Nov. 1950).

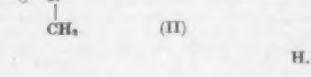
One of the nitration products obtained by refluxing 3:3′-dichloroazoxybenzene with conc. nitric acid, an acidic, water-soluble substance, m.p. 116°c., previously thought to be 3:3′-dichloro-5-nitroazoxybenzene (Bigelow and Steeves, *ibid.*, 17 B, 160 (1939)) has been shown to be 3-chloro-2:4:6-trinitrophenol. A possible mechanism of the reaction is suggested. W. K. R.

Labelled Carcinogenic Azo Dye— 3′-Methyl(^{14}C)-4-dimethylaminoazobenzene. D. A. Salzberg, W. Nye, and A. C. Griffin. *Arch. Biochem.*, 27, 243, 244 (1950): *Chem. Abs.*, 45, 356 (10th Jan. 1951).

Bromobenzene (11 g.) was treated with Mg (1·6 g.) to form C_6H_5·Mg·Br, which was then exposed to CO_2 containing 1·0 millicuries of $^{14}CO_2$, resulting in carboxy-labelled benzoic acid. This was esterified with methanol and nitrated to give a 50% yield of methyl *m*-nitrobenzoate. To this compound (2 g.) in diethylene glycol (12 ml.) were added 85% hydrazine (3 ml.) and KOH (2 g.) in conc. aq. soln. After 10 min. reflux, steam distillation resulted in a 30% yield of *m*-toluidine. This was diazotised and coupled with dimethylaniline to yield 0·4 g. 3′-methyl-4-dimethylaminoazobenzene (0·4 g.) containing 0·2 millicurie of ^{14}C activity. C. O. C.

Derivatives of Benzthiazole[— Azo Dye]. K. Baker and H. E. Fierz-David. *Helv. Chim. Acta*, 33, 2011–2018 (Dec. 1950).

The reaction between dimethylaniline and sulphur, when conducted at a lower temperature for a shorter period than in previous recipes, has afforded an increase of yield of 2-thio-3-methyl-2:3-dihydrobenzthiazole (I) from 6·7 to 34·0%; the addition of zinc chloride and iodine does not improve the yield. The reaction between diethylaniline and sulphur affords only tar, but *N*-methyl-*N*-benzylaniline reacts with sulphur to give a 31·3% yield of 2-phenylbenzthiazole. Condensation of (I) first with hydrazine and then with alcoholic *p*-nitrobenzaldehyde yielded (II), which on reduction with Zn dust and NaOH followed by sulphonation gave a direct azo dye, redder than Naphthamine Yellow NN but of lower alkali and light fastness.



(II)

H. H. H.

Orange GG. J. Rais. *Chem. Obzor.*, **25**, 97–101 (1950): *Chem. Abs.*, **44**, 11,099 (25th Nov. 1950).

It is possible to increase the yield of Orange GG by about 30% if a little of the disodium salt of R acid is added to the passive component. Thus the final product contains some Orange R, which up to a certain point increases the strength of the yellow colour in Orange GG; if this point is exceeded, then it is impossible, in spite of the great strength of the colour of Orange GG, to correct the hue without loss of brilliancy. The exhaustion of the dyebaths and the extinction curves in relation to the amount of added salt of R acid are discussed, and the properties of the pure salts of G and R acids are described. C. O. C.

Influence of Sulpho Groups on the Degree of Dissociation of the Hydroxyl Group in Naphtholsulphonic Acids. H. Zollinger and W. Büchler. *Helv. Chim. Acta*, **33**, 2002–2010 (Dec. 1950).

The dissociation constants of the hydroxyl groups in the 1:3-, 1:4-, 1:5-, 2:6-, and 2:7-naphtholsulphonic acids and also in the 1:3:7- and 7:3:1-hydroxysulphonaphthyl *p*-toluenesulphonates have been determined by potentiometric titration, and the effect of the sulpho group and of its position on the acid strength of the hydroxyl group are discussed. The sulpho group has essentially an electrostatic influence, and both a charge and a dipole effect can be distinguished, of which the latter is the stronger in the examples chosen. Theoretical calculation of the dissociation constants proved to be impossible owing to the non-homogeneity of the electric field within the molecular region. H. H. H.

Bleaching of Malachite Green. W. Jost and K. G. Fahlbusch. *Z. anorg. Chem.*, **262**, 79, 80 (1950): *Chem. Abs.*, **44**, 10,523 (25th Nov. 1950).

The quantum yield for bleaching in 10^{-5}M. soln. by polychromatic light was at pH 3 (H_2SO_4) 5×10^{-4} in H_2 or N_2 and 3×10^{-4} in O_2; at pH 5·3–6·2 (phosphate buffers) it was $3–5 \times 10^{-5}$. Addition of H_2O_2 greatly increased the quantum yield. C. O. C.

Ionisation of Triarylcarbinol Indicators in Sulphuric Acid–Water Mixtures. M. A. Murray and G. Williams. *J.C.S.*, 3322–3326 (Nov. 1950).

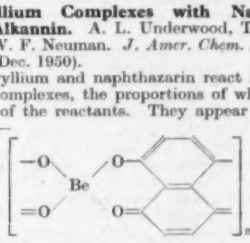
The extent of ionisation of the triarylcarbinol indicators — 4:4′:4″-tris(trimethylammonium)triphenylcarbinyl methyl ether triperchlorate, 4:4′:4″-tris(trimethylammonium)triphenylcarbinol triperchlorate, 4 : 4′ - bis - (trimethylammonium)triphenylcarbinyl methyl ether diperchlorate, and 4:4′:4″-trinitrotriphenylcarbinol— in 90–65% sulphuric acid has been measured with a photoelectric absorptiometer; the above indicators carry fully ionised trimethylammonium groups as nuclear substituents. Westheimer and Kharasch's measurements (*J. Amer. Chem. Soc.*, **68**, 1871 (1946)) on the ionisation of trinitrotriphenylcarbinol in 95–80% sulphuric acid are confirmed. The representation of the ionisation by the equation—

$$Ar_3C{\cdot}OH + 2H_2SO_4 \rightleftharpoons Ar_3C^+ + OH_3^+ + 2HSO_4^-$$

is experimentally established only for nearly anhydrous sulphuric acid media. H. H. H.

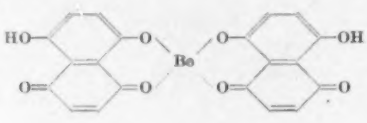
Beryllium Complexes with Naphthazarin and Alkannin. A. L. Underwood, T. Y. Toribara, and W. F. Neuman. *J. Amer. Chem. Soc.*, **72**, 5597–5602 (Dec. 1950).

Beryllium and naphthazarin react in solution to form two complexes, the proportions of which depend on the ratio of the reactants. They appear to have the structures—



(the portion in brackets being repeated to form a polymer chain)

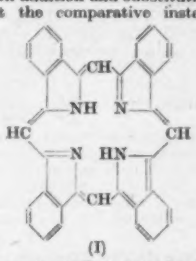
and



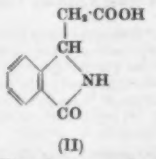
The beryllium complexes of alkannin appear to be analogous to those of naphthazarin. C. O. C.

Phthalocyanines and Related Compounds. XX— Further Investigations on Tetrabenzporphin and Allied Substances. R. P. Linstead and F. T. Weiss. *J.C.S.*, 2975–2981 (Nov. 1950).

The preparation and purification of tetrabenzporphin (I) and of its zinc compound from 3-carboxymethylphthalimidine (II) and zinc acetate is described. Zinc tetrabenzporphin is among the most highly coloured of organic compounds with $\lambda_{max} = 6320$ A. in the red, where $\log_{10}\varepsilon = 5{\cdot}18$. In the sulphonation of phthalocyanine and other macrocyclic pigments, it is shown that the sulphonated pigments are more easily oxidised than the parent substances; the sulphonation of phthalocyanine occurs in the 4-position, although this may not be the only position attacked. In the halogenation of tetrabenzporphin, both addition and substitution of halogen can be realised, but the comparative instability towards halogenating



(I)



(II)

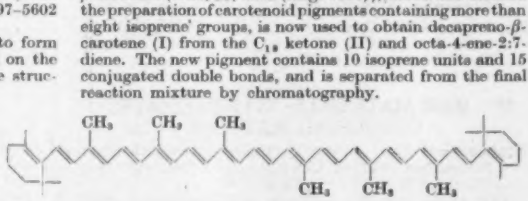
agents is attributed to the instability of compounds substituted on the linking (*meso*) carbon atoms. H. H. H.

Phthalocyanines and Related Compounds. XXI— Oxidation of Phthalocyanine, Tetrabenzporphin, and Allied Substances. R. P. Linstead and F. T. Weiss. *J.C.S.*, 2981–2987 (Nov. 1950).

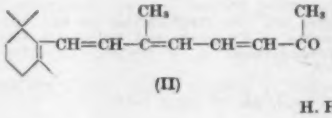
Oxidation of macrocyclic pigments of the phthalocyanine type in suspension in sulphuric acid by ceric sulphate is found to occur stepwise, the first stage being the formation of a purple or brown intermediate, which subsequently either undergoes hydrolysis, as in the case of phthalocyanine, or further oxidation, as for pigments containing methin links. The initial reaction involves addition of oxygen, probably in the form of hydroxyl groups, and not dehydrogenation. The intermediate oxidation product from tetrabenzporphin is highly oxygenated, and some fixation of sulphuric acid occurs. The quantitative oxidation of pigments gives results which indicate divergence from simple stoichiometric relationships where the pigments contain many methin links. H. H. H.

Synthesis of Carotenoids. VI — Synthesis of a Homologue of β-Carotene with Fifteen Conjugated Double Bonds— Decapreno-β-carotene. P. Karrer and C. H. Eugster. *Helv. Chim. Acta*, **34**, 28–33 (Feb. 1951).

The authors' synthetic procedure for the preparation of β-carotene (*ibid.*, **33**, 1172 (Aug. 1950)), which has enabled the preparation of carotenoid pigments containing more than eight isoprene groups, is now used to obtain decapreno-β-carotene (I) from the C_{19} ketone (II) and octa-4-ene-2:7-diene. The new pigment contains 10 isoprene units and 15 conjugated double bonds, and is separated from the final reaction mixture by chromatography.



(I)



(II)

H. H. H.

Carotenoids from *Cladophora glomerata*. P. Karrer and G. Taffi. *Helv. Chim. Acta*, **33**, 2211–2212 (Dec. 1950).

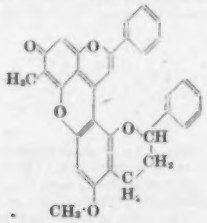
The following carotenoids are shown to exist in *Cladophora glomerata*— β-carotene, xanthophyll, epoxyxanthophyll, violaxanthin, and perhaps also auroxanthin, the isomeride of violaxanthin. H. H. H.

Some Esters of Antheraxanthin, Epoxyxanthophyll, and Trollichrome. P. Karrer, E. Leumann, and W. Eichenberger. *Helv. Chim. Acta*, **33**, 2213–2215 (Dec. 1950).

The diacetate, dipalmitate, and distearate of antheraxanthin, the dipalmitate and distearate of epoxyxanthophyll, and an acetate of trollichrome are described. These esters possess absorption spectra in which the positions of the absorption maxima are almost identical with those of the unacetylated carotenoids. Chloroform containing hydrochloric acid transforms the epoxy esters into the corresponding furanoid oxides, as in the case of the epoxy pigments. H. H. H.

Pigments of "Dragon's Blood" Resins. III—Constitution of Dracorubin. A. Robertson, W. B. Whalley, and J. Yates. *J.C.S.*, 3117–3123 (Nov. 1950).

By degradation and synthesis, dracoic acid, an oxidation product of dracorubin (I), is shown to be (−)-7-hydroxy-5-methoxyflavan-8-carboxylic acid. The formula given for draconal, another oxidation product of I, indicates structure I for dracorubin. The syntheses of 5:7-dihydroxyflavan-6-carboxylic acid and the dimethyl ether of its ester are described, and the natural flavanone alpinetin is shown to be 7-hydroxy-5-methoxyflavanone.

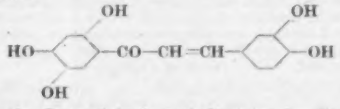


(I)

H. H. H.

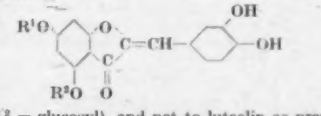
Anthochor Pigments. VI—Pigments of *Coreopsis Stillmanii*. VII—Pigments of Yellow *Antirrhinum majus*. M. K. Seikel and T. A. Geissman. *J. Amer. Chem. Soc.*, **72**, 5720–5724; 5725–5730 (Dec. 1950).

VI—The anthochor pigment of *Coreopsis Stillmanii*, stillopsin, is a hexoside of 3:4:2′:4′:5′-pentahydroxychalkone—



and is the first polyhydroxychalkone to be discovered among plant pigments.

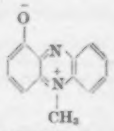
VII—The yellow of *Antirrhinum majus* is due mainly to aureusin, a glucoside of 3′:4′:4:6-tetrahydroxybenzylidenecoumaranone—



(R^1 or R^2 = glucosyl), and not to luteolin as previously reported. In addition a flavonone pigment which is a glycoside of naringenin is present. C. O. C.

Dipole Moment of Pyocyanine. K. A. Jensen and C. H. Holten. *Acta Chem. Scand.*, **3**, 1446–1447 (1949): *Chem. Abs.*, **44**, 10,409 (25th Nov. 1950).

Measurement of the dipole moment of the blue colouring matter, pyocyanine, of the bacillus *Serratia marescens* shows that the resonance structure—



is the only one that is important. C. O. C.

Formation of Tyrosine Melanin—II. G. R. Clemo and F. K. Duxbury. *J.C.S.*, xiv (Dec. 1950).

Corrections to previous paper, *J.C.S.*, 1795–1800 (July 1950): *J.S.D.C.*, **66**, 598 (Nov. 1950).

Colouring Matters of the Aphididæ. IV—General Survey of the Occurrence of the Aphins. H. Duewell, J. P. E. Human, A. W. Johnson, S. F. MacDonald, and A. R. Todd. *J.C.S.*, 3304–3309 (Nov. 1950).

A survey is made of the distribution of aphins and other colouring matters in some 20 species of Aphididæ, and a rapid method is outlined for the detection of aphins in the presence or absence of non-aphin colouring matters. Further procedures have also been developed for the detection and separation of the individual aphins from complex pigment mixtures. The isolation of aphins from *Aphis sambuci* L., *Eriosoma lanigerum*, *Myzus cerasi* F., *Sappaphis pyri* (Fonsc.), and *A. philadelphi* is described. H. H. H.

PATENTS

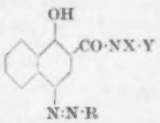
Dialkoxyphenols as Azo Components for Yellow-to-Sepia Diazotypes. General Aniline & Film Corpn., W. H. von Glahn, and L. N. Stanley. *U.S.P.* 2,523,889.

Compounds of formula—

(one of X, Y, and Z = H and the other two = CH_3, C_2H_5, or C_2H_4OH), e.g. pyrogallol 1:3-dimethyl ether, are excellent azo components for producing yellow-to-sepia diazotypes. C. O. C.

1-Hydroxy-2-naphthamide Colour Couplers. Eastman Kodak Co., D. B. Glass, P. W. Vittum, and A. Weissberger. *U.S.P.* 2,521,908.

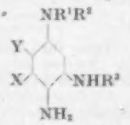
Compounds of formula—



(R = Ar of the benzene series; X = H or Alk; Y = Ar of the benzene series or aralkyl), e.g. 1-hydroxy-4-phenylazo-4′-*p-tert.*-butylphenoxy-2-naphthanilide, yield blue–green dyes on oxidised development. C. O. C.

Colour Formers for Yellow Azine Dyes. General Aniline & Film Corpn., D. E. Sargent, and R. C. Gunther. *U.S.P.* 2,522,802.

Permanent yellow dye images can be produced simultaneously with cyan and magenta azine dyes by colour-forming development with a compound of formula—



(R^1 = H or Alk; R^2 = Alk, hydroxyalkyl, sulphoalkyl, or an aromatic radical; R^3 = aliphatic or aromatic radical; X and Y = H, Alk, COOH, or SO_3H), e.g. 6-phenylamino-4-methylaminometanilic acid, in presence of a ketomethylene compound, e.g. α-benzoylacetanilide, the pH in the final stages of processing being adjusted by addition of an alkali until the yellow image appears. C. O. C.

Phenolphthalein. Monsanto Chemical Co. and H. R. Gamrath. *U.S.P.* 2,522,939.

When condensing phthalic anhydride and phenol in presence of $ZnCl_2$ addition of a little thionyl or sulphonyl chloride or chlorosulphonic acid results in exceptionally high and pure yields.

U.S.P. 2,522,940.

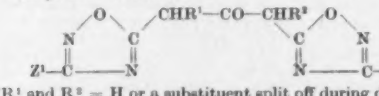
An arylsulphonyl chloride has the same effect. C. O. C.

Coupling Component for Diazotype Light-sensitive Layers. General Aniline & Film Corpn., F. W. Neumann, and G. W. Pedlow, Jr. *B.P.* 648,585.

Use of 2:2′:4:4′-tetrahydroxydiphenyl as the azo coupling component with *p*-phenylenediamine diazo compounds yields deep brown images of excellent fastness to light and water. C. O. C.

Colour Couplers. Gevaert Photo-Producten N.V. and P. L. J. R. Merckx. *B.P.* 648,907.

Compounds of formula—



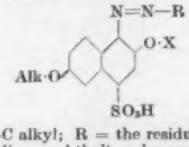
(R^1 and R^2 = H or a substituent split off during development, e.g. Hal, Ar, SO_2H; Z^1 and Z^2 = organic radicals), in which one or both of the H atoms of the CHR^1·CO·CHR^2 group may be replaced by a substituent split off during development, are used as colour couplers in conjunction with a primary aromatic amino developer. C. O. C.

Metachrome Dyes—*O*-Acylated Monoazo Compounds. Ciba Ltd. *B.P.* 648,715.

oo′-Dihydroxymonoazo dyes unsuitable for metachrome dyeing may be rendered suitable by *O*-acylation. Such acylation, as described in *B.P.* 636,681 (cf. J.S.D.C., **66**, 451 (Aug. 1950)), was formerly conducted in non-aqueous media, but it can be achieved under alkaline aqueous conditions by using as acylating agents derivatives of benzoic acid free from solubilising groups, e.g. benzoyl chloride or a nitrobenzoyl chloride. Thus the monoazo compound 1-amino-2-naphthol-4-sulphonic acid →1-phenyl-3-methyl-5-pyrazolone is acylated with benzoyl chloride at 15–20°c. in presence of aq. NaOH and sodium acetate. The product is a bluish-red metachrome dye. E. S.

Metalliferous Monoazo Acid Dyes. Ciba Ltd. *B.P.* 648,364.

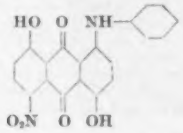
Acid dyes are made by treating with metallic (especially chromium) compounds monoazo dyes of formula—



(X = H or low-C alkyl; R = the residue of a pyrazolone or an *ortho*-coupling naphthol) under such conditions that where X is alkyl it is split off during the metallisation. Thus 6-methoxy-2-naphthol is treated with cold nitrous acid to give the 1-nitroso compound, which reacts with sodium bisulphite to give the 1-hydroxylamino-4-sulphonic acid. Further reaction with dil. H_2SO_4 gives 1-amino-6-methoxy-2-naphthol-4-sulphonic acid. This is diazotised in presence of $CuSO_4$ and coupled under alkaline conditions with β-naphthol; the resulting monoazo compound is refluxed with aq. CrF_3 to give a greenish-blue acid dye. E. S.

Anthraquinonoid Dyes for Cellulose Esters and Ethers. British Celanese Ltd. *B.P.* 646,459.

Blue dyes for cellulose acetate fast to gas fumes are made by condensing 4:8-dinitroanthrarufin or 4:5-dinitrochrysazin with a primary amine. Thus the dye—



is prepared by refluxing 4:8-dinitroanthrarufin (1 mol.) with aniline (15 mol.) containing a little water. The product is separated by adding ethanol and HCl. R. K. F.

Partly Methylated 1:4:5:8-Tetra-aminoanthraquinone. British Celanese Ltd. *B.P.* 647,325.

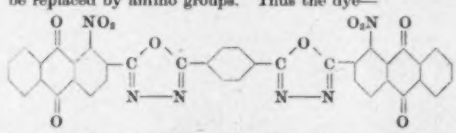
In making 1:4:5:8-tetra-aminoanthraquinone by nitrating, reducing, and hydrolysing acylated crude 1:5- and 1:8-diaminoanthraquinones, β-nitration is avoided and the purity of the final product improved by nitrating in presence of boric acid. Methylation to give a dye whose hue on cellulose acetate changes only slightly in artificial light is carried out simultaneously with hydrolysis by adding methanol to the solution of diacylated tetra-aminoanthraquinone in 98% H_2SO_4 and heating at 130–135°c. R. K. F.

Sulphamyl-acylaminoanthraquinones—Vat Dyes. Ciba Ltd. *B.P.* 646,950.

Vat dyes are produced by treating an aminoanthraquinone having a β-sulphamyl group with an acylating agent. Thus 1-aminoanthraquinone-6-sulphodimethylamide, prepared by treating 1-nitroanthraquinone-6-sulphonic acid with $POCl_3$ and PCl_5 followed by dimethylamine and finally reducing with sodium hydrosulphide, is refluxed in chlorobenzene with *p*-chlorobenzoyl chloride. R. K. F.

Anthraquinone-1:3:4-Oxadiazoles—Vat Dyes. du Pont and F. B. Stilmar. *U.S.P.* 2,511,018, *U.S.P.* 2,511,019.

Red vat dyes are made by condensing a 1-nitroanthraquinone-2-carboxyhydrazide with oxalyl chloride or a benzene-, naphthalene-, or di- or ter-phenyldicarboxylic chloride or nuclear-halogenated derivative thereof, and cyclising the hydrazide groups. The nitro groups may be replaced by amino groups. Thus the dye—

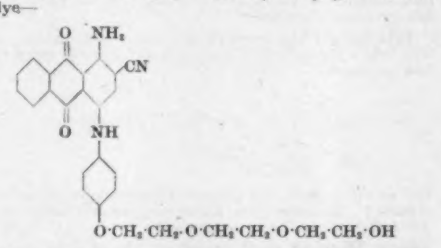


is prepared by treating 1-nitroanthraquinone-2-carboxyhydrazide (2 mol.) with terephthaloyl chloride (1 mol.) in nitrobenzene at 165–170°c. for 2 hr. The product after filtering off is cyclised by refluxing in nitrobenzene with thionyl chloride. The nitro groups may be replaced by amino groups to give a dye of similar properties by passing ammonia through a suspension in nitrobenzene at 170–200°c. R. K. F.

1-Amino-2-cyano-4-*p*-polyethoxyphenylamino-anthraquinones—Dyes for Cellulose Esters and Superpolyamides. Eastman Kodak Co., J. B. Dickey, and A. Loria. *U.S.P.* 2,510,088.

Blue dyes, fast to light and gas fumes, for cellulose acetate and nylon are prepared by condensing cuprous cyanide in presence of quinoline with a 1-amino(or subst. amino)-2-bromoanthraquinone containing in position 4 a phenylamino group in which the phenyl group carries *para* to the –NH– a group of formula—

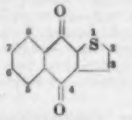
$-O\cdot CH_2\cdot CH_2\cdot[O\cdot CH_2\cdot CH_2]_n\cdot OH$ or $-[O\cdot CH_2\cdot CH_2]_m\cdot O\cdot R$ (n = 1–3; m = 2 or 3; R = CH_3 or C_2H_5). Thus the dye—



is made by refluxing the corresponding 2-bromo compound, prepared by treating 1-amino-2:4-dibromoanthraquinone with *p*-β-hydroxy-β-ethoxy-β-ethoxyethoxy-aniline, in quinoline with cuprous cyanide, and isolated by pouring into dil. HCl. R. K. F.

Monohalogenothiophanthraquinones. du Pont, L. F. Fieser, and H. E. Schroeder. *U.S.P.* 2,513,790.

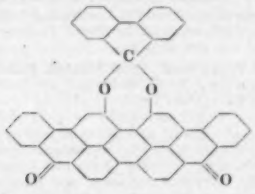
Derivatives of thiophanthraquinone—



carrying a bromine or chlorine atom at position 2, 3, 5, 6, 7, or 8 are prepared by reducing a halogen-substituted 2-*o*-thienoylbenzoic acid to the corresponding thenyl compound, cyclising in acetic anhydride to the 4-acetoxythiophanthren, and finally oxidising to the monohalogenothiophanthraquinone. Thus 6-chloro-2-(2′-thienoyl)-benzoic acid is reduced by boiling with ammonia and zinc dust. The product is refluxed in a mixture of acetic anhydride, acetic acid, and fused $ZnCl_2$. The resulting 4-acetoxy derivative is then oxidised in acetic acid containing chromium trioxide, and the resulting 5-chlorothiophanthraquinone purified by vatting. R. K. F.

Dibenzanthrone Derivative. General Aniline & Film Corpn., F. Max, and W. Schmidt-Nickels. *U.S.P.* 2,515,723.

16:17-Dihydroxydibenzanthrone (1 mol.) is heated with 9:9-dichlorofluorene (3–4 mol.) and anhyd. sodium acetate (6 mol.) in nitrobenzene at 200–205°c. for 25 hr. The resulting product—



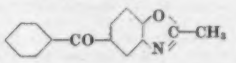
is a blue vat dye. R. K. F.

Copper Phthalocyanine Polymers. American Cyanamid Co., E. L. Kropa, and J. J. Roemer. *U.S.P.* 2,513,098.

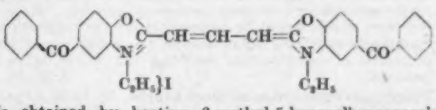
Polymers containing > 1 phthalocyanine ring per mol. are prepared by treating an *o*-dichlorostyrene (containing the 2 Cl *ortho* to each other) with cuprous cyanide. The resulting pigments are only partly soluble in H_2SO_4. Thus *o*-dichlorostyrene is heated at 210–250°c. with cuprous cyanide in pyridine. R. K. F.

Cyanine Dyes. du Pont and J. C. Firestine. *U.S.P.* 2,521,705.

2-Methyl-5-benzoylbenzoxazole—



is readily prepared by treating a solution of *p*-hydroxybenzophenone with nitric acid, and reducing the resulting nitro-*p*-hydroxybenzophenone with metal dust and acid to the amino compound, which is simultaneously or subsequently condensed with acetic anhydride to yield the final product. This on treatment with an alkyl or aralkyl salt is converted into a *cyclo*ammonium or quaternary salt, which on treatment with a reactive heterocyclic nitrogen salt forms a mono- or poly-methin dye. Thus the dye—



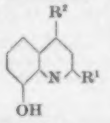
is obtained by heating 2-methyl-5-benzoylbenzoxazole with diethyl sulphate for 2 hr. at 130–140°c. Cooling the melt, treating with pyridine and ethyl orthoformate under reflux, and then adding excess sat. aq. KI to the solution precipitates the dye as red needles. C. O. C.

Polymethin Dyes. Eastman Kodak Co. and V. P. Pittman. *U.S.P.* 2,521,944.

Dicarboxyamine dyes containing two pentamethin chains, the chains connected by a bond and each chain ending in a heterocyclic group containing an auxochromic N atom, are obtained by condensing a *cyclo*ammonium quaternary salt containing a reactive methyl group with a β-formyl-β-halogenoacrylic acid containing in the α-position an ionisable group other than a halogen atom. C. O. C.

Magenta Phenazonium Dyes from 8-Hydroxyquinoline Couplers. General Aniline & Film Corpn. and R. F. Coles. *U.S.P.* 2,524,725.

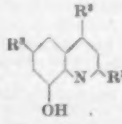
Compounds of formula—



(R^1 = an electronegative group, preferably Hal or a mononuclear carbocyclic aromatic radical; R^2 = H, COOH, or a carbamyl group containing an alkyl chain of > 9 C) used as colour couplers with an *o*-phenylenediamine developer yield magenta phenazonium dyes. C. O. C.

Magenta Phenazonium Dye Images. General Aniline & Film Corpn., V. Tulagin, and R. F. Coles. *B.P.* 647,638, *U.S.P.* 2,524,741.

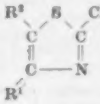
Magenta images are obtained by developing an exposed light-sensitive silver salt emulsion coating with a 2:4-diaminoaniline developer in presence of a colour former of formula—



(R^1 = H, Alk, or a mononuclear carbocyclic aromatic radical; R^2 = H, COOH, or CO·NH·R^4; R^3 = Hal, NO_2, SO_3H, or AsO_3H_2; R^4 = sulphonated aliphatic radical of > 9 C or a sulphonated aromatic radical substituted by an aliphatic radical of > 9 C), e.g. 6-sulpho-8-hydroxyquinoline. C. O. C.

2-Methylthiazoles as Intermediates for Cyanine Dyes. du Pont and E. B. Middleton. *U.S.P.* 2,524,674, *U.S.P.* 2,524,675.

2-Methylthiazoles, which contain a furyl nucleus in the 4-position and which may be substituted in the 5-position, can be converted into *cyclo*ammonium or quaternary salts by treatment with a salt-forming compound. The reactive methyl group α to the heterocyclic N atom enables them to condense to form cyanine dye intermediates of formula—



(R^1 = a furan nucleus attached α to its O atom; R^2 = a similar furan nucleus or H, hydrocarbon radical, or carbalkoxy group). C. O. C.

Powdered Metal. G. R. Smith, G. D. Todd, Jr., and W. S. Lindley. *U.S.P.* 2,522,546.

Metal powder is produced by mixing a finely ground carbide with molten metal, cooling till solid, and then treating with water. C. O. C.

Leafing Agents for Metal Flake Pigments. Reynolds Metals Co., F. B. Rethwisch, and G. M. Babcock. *U.S.P.* 2,522,538.

Aliphatic amines containing > 13 C and of mol. wt. 185–300 give better leafing than stearic acid and like leafing agents, and the product has a better colour. In

addition, the product mixes and disperses more readily in the usual vehicles, and there is less tendency towards surface skin formation in paints in which they are used. C. O. C.

Bleaching Barytes. C. K. William & Co. and T. K. Stinson. *U.S.P.* 2,522,971.

Fluorspar-free barytes is ground, treated with a strong oxidising agent, and then given the usual sulphuric acid treatment. The oxidising treatment converts the galena present to lead sulphate, the pyrites to readily soluble ferric sulphate, and organic matter to CO and CO_2. The final product is a purer white than that prepared without the oxidation treatment. C. O. C.

Orange–Dark Red Cadmium–Selenium Pigment. Ferro Enamel Corpn., O. O. Kenworthy, E. H. Goda, and J. D. Golland. *U.S.P.* 2,523,119.

A highly saturated cadmium–selenium pigment, stable up to 1800°F. and insoluble in water and organic solvents, is produced by calcining cadmium sulphide, selenium, and a claylike substance. C. O. C.

Carbon Black. Phillips Petroleum Co. *B.P.* 649,091.

A method is described for producing a high yield of furnace black having properties similar to those of the normal low-yield, hard blacks. C. O. C.

γ-Ferric Oxide. General Electric Co. Ltd., D. M. Dovey, R. C. Chirnside, and H. P. Rooksby. *B.P.* 648,494.

Finely divided γ-ferric oxide of uniform particle size is obtained by heating finely divided iron oxalate or formate at such a temperature that only Fe_3O_4 is produced, this then being converted into Fe_2O_3 by controlled oxidation. C. O. C.

Magnesium Oxide. Permanente Metals Corpn., A. W. Vettel, A. Covello, and F. A. Demaestri. *B.P.* 648,315.

Describes the production of MgO by treating dolomite with seawater. C. O. C.

Grey Pigments for use in Ceramics and in Enamelling. Société des Usines Chimiques Rhône-Poulenc. *B.P.* 648,559.

Mixtures of 1–65% by weight of MoO_2, 35–99% of Al_2O_3, and 0–40% of ZnO or SnO_2 yield deep bluish to light greys, depending primarily on the proportion of MoO_2 present. C. O. C.

Zinc Oxide. Union Oxides (Proprietary) Ltd. *B.P.* 647,887.

Zinc oxide of high purity and very even crystalline structure, which is pure white, is obtained by passing a continuous current of oxidising gases over molten zinc or zinc alloy maintained at approx. 10°c. below b.p. C. O. C.

Dithionate Process for producing Zinc Hydroxide from Oxidised Zinc Ores. U.S. Secretary of the Interior, S. F. Ravitz, and A. E. Back. *U.S.P.* 2,522,537.

An aqueous slurry of the oxidised ore is treated with SO_2 so as to dissolve the Zn as zinc dithionate and zinc sulphate. The undissolved materials are filtered off, and the liquor is treated with calcium dithionate to convert the zinc sulphate to the dithionate. The precipitated calcium sulphate is removed, and the liquor treated with calcium hydroxide. The zinc hydroxide is filtered off, and the calcium dithionate filtrate is used for treating another batch of sulphate-containing zinc solution. C. O. C.

Diazotype Photoprinting Materials. General Aniline & Film Corpn. and H. C. Unkauf. (IX, p. 167.)

V—PAINTS; ENAMELS; INKS

PATENTS

Heat-resistant Paint. Socony-Vacuum Oil Co. Inc. and P. Robey. *U.S.P.* 2,524,357.

In the films formed by paints based on colloidal silica, the non-leafing particles of a metallic pigment are distributed evenly throughout the whole thickness of the film, reinforcing it and being much stronger than when leafing pigments are used. Addition of phosphoric acid or a C_{1-4} alkyl partial ester of phosphoric acid increases the adhesion of the silica vehicle for the metal pigment. C. O. C.

Pigmented Coating Compositions containing Liquid Alkyl Polysiloxane. General Electric Co. and C. M. Sage. *U.S.P.* 2,523,065.

Coating compositions, having good pigment antiflotation properties, reduced tendency to show "silking" and "flow" lines, and yielding smooth surfaces which do not show "orange peel", are obtained by adding 0·00001–0·001% by weight on the total resin and pigment content of a liquid alkyl polysiloxane to pigmented resin coating compositions. C. O. C.

Hot-melt Coating Composition. British Celanese Ltd. (III, p. 157.)

VI—FIBRES; YARNS; FABRICS

Problem of Cellulose Modifications. A. Sippel. *Kolloid-Z.*, **119**, 42–45 (Oct.) and 169 (Dec. 1950).

A periodic tension-free model is proposed for cellulose of period two unit molecules of cellobiose, which, in the form adopted, possesses a cellobiose unit length of ~ 9·8 A., and permits the explanation of the limited extensibility of cellulose fibres. H. H. H.

New Concepts in the Structure of Cellulose and its Derivatives. F. Happey. *J. Textile Inst.*, **41**, T 454 (Nov. 1950).

Correction to previous paper, *J. Textile Inst.*, **41**, T 381–T 403 (Oct. 1950): J.S.D.C., **67**, 90 (Feb. 1951).

Cultivation of Coloured Cotton. A. Lübke. *Textil-Praxis*, **5**, 767–769 (Dec. 1950).

Review of attempts to cultivate coloured cottons. So far the production of colour has been attended by great reduction in the quality of the fibres. Russian experiments in this field are not original. F. A.

Studies of Worn and Aged Cotton Fabrics. M. E. Murdison and J. S. Roberts. *J. Textile Inst.*, **41**, T 429–T 445 (Nov. 1950).

Damage in cotton subjected to heat and weathering effects, in aged cotton, and in worn discarded cotton is measured by tensile strength, cuprammonium fluidity, and Congo Red staining. Twenty photomicrographs are shown. Oven drying at 100°c. tenders the material and progressively damages the cuticle, whereas up to 100 dryings at 60°c. and controlled ironings give much smaller breakdown. Oxidative degradation due to sunlight, Weatherometer, or chlorine bleaching is accompanied by visual surface damage to the cuticle, but a clearly different injury results from purely mechanical damage. Normal wear and washing of tea-towels are more damaging than individual launderings without use, and breakdown characteristics of very old fabrics (500 years) are not distinct from those of new material. For examination of progressive tendering, or comparison of wearing qualities of finished garments, the Congo Red test is of most value. J. W. B.

X-Ray Study of the Soda Celluloses of Jute treated with Caustic Soda at Room Temperature. R. R. Mukherjee and H. J. Woods. *J. Textile Inst.*, **41**, T 422–T 428 (Nov. 1950).

When jute is treated without tension at 20°c. in NaOH solutions of > 10% concentration there is a complete transformation of the crystalline material to soda cellulose. Removal of the alkali by washing at room temperature leads to partial conversion to the native material, the presence of which is clearly indicated by X-ray photographs. This partial mercerisation is ascribed to the lignin, which, though it is extra-crystallite in location and does not prevent entry of alkali into the crystalline regions, acts as a cross-linking agent in the non-crystalline portion and thus stabilises the system. To obtain strong enough bonds to withstand NaOH of mercerising strength, condensation is suggested between reactive OH groups in lignin and primary alcoholic groups in cellulose. Even a few such bonds would be responsible for the difficulty of completely removing lignin and for the persistence of the inhibition of mercerisation until the bulk of the lignin is removed. J. W. B.

Frictional Properties of Wool Fibres. K. R. Makinson; G. King. *J. Textile Inst.*, **41**, T 493–T 494 (Dec. 1950).

In reply to a letter by King (*ibid.*, **41**, T 135 (April 1950)) Makinson considers that the variation of normal force per unit length with the total load between fibre and cylinder is not negligible, and that King's deduction that

the coefficient of friction increases as the area is reduced is not valid.

A letter by King follows in which, in reply to the first point only, his original argument is reiterated and elaborated. J. W. B.

Supercontraction of Wool Yarn. J. Menkart and W. Mitchell. *J. Textile Inst.*, **41**, T 491–T 492 (Dec. 1950).

An attempt is made to correlate alkali damage and supercontraction of yarns in boiling sodium bisulphite solution. Undamaged yarn contracts as much as the constituent fibres and alkali-damaged yarns fail to contract, but in intermediate stages the yarn is still largely or fully supercontracted simply through the influence of the few non-damaged fibres, which pull the whole lot together. Thus intermediate values cannot be obtained and measurements on yarn do not give a dependable estimate of the supercontraction characteristics of the constituent fibres. J. W. B.

Cellulose Fibres. XIII—Xanthation of Degraded Celluloses. K. Lauer and O. Pauer. *Kolloid-Z.*, **119**, 155–156 (Dec. 1950).

The heterogeneous xanthation of acid- or alkali-degraded celluloses of various degrees of polymerisation from normal prepiened celluloses gives compounds whose maximum γ-numbers bear a linear relationship to the swelling values of the celluloses. This relationship does not hold, however, for grades of low expression. Fibre-free viscoses with γ-numbers of ~ 50 have thus been obtained from degraded celluloses independently of the method of xanthation employed. H. H. H.

Relationship between Structure and Creasing Properties of Viscose Rayon—I. E. Elöd and M. Kramer. *Textil-Praxis*, **6**, 52–56 (Jan. 1951).

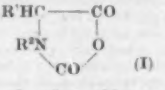
The literature is reviewed and some experimental evidence presented to relate the influence of variations during the manufacture of viscose on its creasing properties. Effect of variations in drawing, stretching, atmospheric moisture, denier, type of cross-section, α-cellulose and alkali content of the spinning mass, degree of crystallinity, and also various forms of aftertreatment are discussed. Although careful control of spinning conditions is advantageous, it is not to be anticipated that marked improvement in crease-resisting properties can be obtained by this alone. Excess of synthetic resin used in subsequent crease-resisting results in a decreased resistance to shear and also increased sensitivity to chlorine and washing. B. K.

Optical and X-Ray Investigations of Synthetic Perlon Fibre. E. Kordes, F. Günther, L. Büchs, and W. Göltner. *Kolloid-Z.*, **119**, 23–38 (Oct. 1950).

Like many macromolecular substances, Perlon consists of amorphous and crystalline components, whose ratio depends on manufacturing conditions. Double refraction and X-ray data, obtained from mechanically deformed samples of Perlon, enable conclusions to be drawn concerning changes in fine structure during deformation; both sets of data indicate the same results. It is found that the lattice structure of the crystalline component is independent of the chain-length of the molecule when the lowest degree of polymerisation is considered. In Perlon used for hose, the lactam content may be detected by means of X-rays down to 5–6%. There are 11 illustrations. H. H. H.

Synthetic Polypeptides—I. W. E. Hanby, S. G. Waley, and J. Watson. *J.C.S.*, 3009–3013 (Nov. 1950).

The preparation of several *N*-carboxyamino acid anhydrides (I) is described, and methods are discussed for their polymerisation. In a new general method, now investigated, the anhydrides are polymerised in nitrobenzene solution by amino acid dimethylamides to give



(I)

quantitative yields of polypeptides; copolymers are readily prepared by this method. The following are described—polyglycine, poly-DL-alanine, polysarcosine, poly-L-leucine, poly-DL-valine, and poly-DL-phenylalanine dimethylamides, and the copolymers—glycine–sarcosine, α-amino*iso*butyric acid–DL-phenylalanine, α-amino*iso*butyric acid–DL-valine, glycine–DL-phenylalanine, DL-valine–DL-phenylalanine, and L-leucine–DL-phenylalanine. Copolymers were also made from γ-methyl *N*-carboxy-L-glutamate anhydride. In most cases evaporation of a solution of the polymer gave a film, and extrusion of the solution into water, alcohol, or ether afforded fibres. H. H. H.

PATENTS

Improving the Tensile Strength, Elasticity, and Fatigue Resistance of Cotton. B. F. Goodrich Co. and J. Compton. *U.S.P.* 2,522,095.

Cotton fibres from unopened bolls are subjected to a tension of 25–75% of their breaking strength while in a moist atmosphere and while retaining their natural moisture content; the tension is then released, and the fibres are dried and allowed to relax. C. O. C.

Organic Polymeric Fibres. American Viscose Corpn. *B.P.* 648,830.

In the manufacture of fibres from solutions of fibre-forming materials, particularly rayon from viscose, more homogeneity as regards crystallinity is obtained by treating the fibres before their final setting with sound waves of frequency 10^3–10^9 cycles/sec. C. O. C.

Viscose Rayon, Films, etc. Industrial Rayon Corpn. and M. D. Rodgers. *U.S.P.* 2,521,748.

Viscose is extruded into an acid coagulating bath, from which it is withdrawn, treated firstly with aqueous acid at 65–100°c. and then with an aqueous oxidising solution, and finally washed. This considerably reduces the processing time between extrusion and final washing, and gives a product of physical and dyeing properties equal or superior in uniformity to those of commercially acceptable standards. C. O. C.

Zein Fibres. U.S. Secretary of Agriculture, C. D. Evans, C. W. Ofelt, and A. K. Smith. *U.S.P.* 2,521,704.

Very flexible low-denier fibres, of strength greater than that of wool, are obtained by spinning an aqueous dispersion of zein of pH 11–12·8 into an acid coagulating bath. C. O. C.

Curing Prolamine Fibres with Aldehyde in Organic Liquid Medium. U.S. Secretary of Agriculture, C. B. Croston, C. D. Evans, L. L. McKinney, and J. C. Cowan. *U.S.P.* 2,524,042.

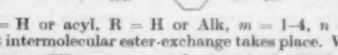
Zein fibres are length-stabilised and given improved resistance to water by means of an acid-catalysed aldehyde cure carried out hot in an anhydrous inert organic solvent. C. O. C.

Artificial Bristles and the like from Proteins. U.S. Secretary of Agriculture, T. L. McMeekin, T. S. Reid, R. C. Warner, and R. W. Jackson. *U.S.P.* 2,521,738.

A protein, e.g. finely divided isoelectric casein, is treated at 80–100°c. with water to form a homogeneous plastic gel. This is then extruded at 95–110°c. into air and passed over rotating drums to stretch the fibre. The fibre is then quickly passed through an aqueous solution of 10% sodium sulphate, 2% formaldehyde, and 0·1% of a wetting agent, and then reeled. Instead of this treatment the fibre may be dried before reeling. C. O. C.

Polyester Fibres. Courtaulds Ltd., A. S. Carpenter, F. Reeder, and E. R. Wallsgrove. *B.P.* 648,513.

The cold-drawing properties of polyquinol sebacate (described in *B.P.* 636,429—cf. *J.S.D.C.*, **66**, 455 (Aug. 1950)) are improved by heating with a preformed polyester obtained by autocondensation of compounds of formula—



(A = H or acyl, R = H or Alk, m = 1–4, n = 0–4) so that intermolecular ester-exchange takes place. W. G. C.

Fibres, Films, etc. of Acrylonitrile-*iso*Butenyl Acetate Copolymers. Eastman Kodak Co. and J. R. Caldwell. *U.S.P.* 2,521,898.

Fibres, Films, etc. of *N*-Fluoroacrylamide Copolymers. Eastman Kodak Co., H. W. Coover, Jr., and J. B. Dickey. *U.S.P.* 2,521,902.

N-Fluoroacrylamides are obtained by treating an acryloyl chloride or a β-chloropropionyl chloride with a primary or secondary fluoroalkylamine in presence of an

organic tertiary base. They readily form polymers or copolymers which can be moulded or formed into films. Solutions of the copolymers, especially those with acrylonitrile, are useful for spinning fibres. C. O. C.

Fibres from a Mixture of α-Cyanoacetamide and an Acrylonitrile Polymer. Monsanto Chemical Co. and G. E. Ham. *U.S.P.* 2,522,445.

α-Cyanoacetamide is an excellent solvent for acrylonitrile polymers and copolymers, solutions being suitable for extruding to yield fibres of high tensile strength and desirable elongation. C. O. C.

Crêpe Threads. British Celanese Ltd. *B.P.* 647,608.

A thread of filaments of an organic derivative of cellulose is highly crêpe-twisted, at least the last part of the crêpe twist being inserted while the thread is under the influence of a hot aqueous medium, e.g. wet steam, and under a tension of at least 0·15 g./denier. J. W. B.

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Properties of Wool with particular reference to Scouring. A. C. Goodings. *Amer. Dyestuff Rep.*, **40**, P 55–P 60 (22nd Jan. 1951).

A comparison is made between the difficulty of removing mineral oil from wool by conventional scouring and the ease of removal from silk and nylon. Improved removal from wool is effected by substituting bicarbonate for carbonate in the soap and soda scour. Abnormal behaviour is induced by certain pretreatments; e.g. 90% of applied mineral oil can be removed by normal methods from chlorinated wool, and wool made acid by dyeing, though this does not apply if the fabric is neutralised with ammonia. It is considered that the relation between oil scourability and fibre surface properties, and comparison between those properties in different fibres, can be utilised in elucidating the fundamental structure of wool. J. W. B.

One-step Stripping Process for Wool. M. Harris and L. W. Rainard. *Amer. Wool & Cotton Rep.*, **64**, (34), 13–14, 18 (1950): *J. Textile Inst.*, **41**, A 520 (Nov. 1950).

In the Harristrip process ethylene dibromide is added to the hydrosulphite or sulphoxylate stripping solution, the cross-linkages in the wool thereby being stabilised. The method is cheaper and quicker, and the material remains essentially unchanged in strength after subsequent dyeing. The method does not reduce the dyeings to an absolute white, but it does significantly increase the whiteness, and the treated fibres are firm, strong, and resilient and retain a desirable handle. C. J. W. H.

Certain Questions concerning the Chemistry of Bleaching Cotton and Flax. P. P. Viktorov. *Tekstil. Prom.*, **10**, 24–26 (Aug.), 31–33 (Sept.), and 31–33 (Nov. 1950).

Three questions about textile bleaching are considered. They concern empirical, practical observations that remain without adequate theoretical basis. Firstly, why does green flax fibre, prepared mechanically, respond more readily to the bleaching process than fibre prepared by biochemical treatment? It is found in practice that fabrics made from green flax attain an adequate degree of whiteness after two bleaching cycles in place of the four that are generally required for retted flax; moreover, the finished fabric has a higher wettability. It is also found that for green flax the absorption of active chlorine is more rapid and the loss in tensile strength during bleaching is apt to be greater— it may be as high as 90% if the usual four bleaching cycles are given. It is suggested that one reason for the differences in behaviour is that natural pigments, present in the green flax only, have a catalytic effect on the oxidation of cellulose. The nature of the pigments known to be present (carotenes, xanthophyll, and chlorophyll) is discussed. The content of pigment may be reduced by extraction with 80% aqueous acetone, and it is then found that the rate of chlorine consumption during bleaching is also reduced. It is suggested that in the retting process these pigments undergo polymerisation and oxidative condensation reactions, leading e.g. to rubberlike substances (such as are present in Indian hemp fibre), the presence of which causes lower wettability. It is stated also that the structure of green flax fibre is more open than that of retted flax, and is therefore more accessible to reagents.

The second question concerns the prekiering hypochlorite bleach introduced by Lomanovich in 1927. This process, in which cotton fabric is treated with alkaline hypochlorite soln. prior to kiering, leads to a considerable reduction in the time required in the kier. However, the method was abandoned owing to the suffocating odour arising from the action of the bleaching agent on raw cotton. It was later found that, when the hypochlorite treatment was preceded by a treatment with mineral acid, the disagreeable odour was not produced. When working with the modified process, it was found that the consumption of active chlorine was much greater than for the normal hypochlorite bleach, carried out *after* kiering (reference is made to a works bleaching process consisting in circulating alkaline hypochlorite soln. through the fabric at 70–80°C.). The question now arises of the nature of the substances that react with the active chlorine in a prekiering bleach, and the reasons for the high chlorine consumption. The part played by nitrogenous matter is considered. Samples of cotton fabric, which had been subjected to various treatments including kier-boiling, were treated with hypochlorite; though there was some correlation between their nitrogen contents and the consumption of active chlorine, the results show that the increased consumption in a prekiering bleach must arise largely from non-nitrogenous matter present. The odour produced during bleaching by the original process is attributed to chloroamines, which arise from nitrogenous matter that is readily removed or destroyed by an acid treatment. The non-nitrogenous matter consists in the main of carbohydrates and their derivatives, but determinations of the iodine number of an aqueous acetone extract of the fibre, and also iodine absorption determinations carried out under neutral and alkaline conditions on the fibre itself, led to the conclusion that an unsaturated substance is present. It is thought that the substance concerned is chlorogenic acid.

Thirdly, why does kiering with lime yield a higher degree of whiteness on cotton and linen fabrics than does kiering with caustic soda or with a mixture of caustic soda and sodium carbonate? The poorer white in these latter cases is attributed to absorption by the fibre of some of the colouring matter that forms during the boil and causes the liquor to become brown. The origin of the brown colour has been variously attributed to pectic substances and to nitrogenous matter; for dark-coloured varieties of cotton the pigment itself may be the main source of colour. Chemical analyses are given of linen, kier-boiled with lime and with caustic soda, but they throw no light on the matter. By treating raw cotton with enzymes, and also by electrodialysis, a series of preparations were made containing various amounts of pectic acid. The preparations were then boiled in caustic soda soln., but there was no relation between the brownness of the used liquor and the pectic acid content of the preparation; in particular, a preparation freed from pectic acid in the normal course of analysis was found to give a liquor of the same colour and the same characteristic smell as that obtained from the original raw cotton. Another sample of raw cotton was treated with hypochlorite, under conditions that had no appreciable degrading effect on the cellulose, so that its nitrogen content was reduced to about 7% of the original value. Kiering this with caustic soda yielded a liquor that was not much paler than that obtained from the original cotton. On the above and other evidence it is concluded that the brown colour cannot be attributed in any great degree to the pectic or nitrogenous substances present. It is considered that the colour arising from light-coloured varieties of cotton is to be attributed mainly to carbohydrate constituents, which have hitherto evaded exact analysis. They are extracted, with other materials, from raw cotton by cold 18% NaOH, and the intensity of the colour of successive extracts can be correlated with the dichromate number of the extract. A soln., similar in colour and smell to used kier liquor, can be obtained by boiling invert sugar with caustic soda soln., and this soln. and kier liquor behave in a similar manner on adding mineral acid, when a brown ppt. is formed. A weaker alkali, such as lime, has a milder effect on the

carbohydrate matter and gives kier liquors of a much lighter colour.

It is concluded that the rubberlike substances, supposed to be present in retted flax, and the carbohydrate constituents of raw cotton that largely account for the dark colour of kier liquors should be the subject of further study. A. E. S.

"Danger Zone" of the Chlorine Bleach. E. A. Shilov and A. A. Yasnikov. *Tekstil. Prom.*, **10**, 35–38 (Nov. 1950).

A summary is given of work by Shilov and co-workers on the kinetics of the oxidation of various organic substances by hypochlorite solutions over a wide range of pH. For starch and hydroxy acids, such as lactic and gluconic acids, the relation between reaction rate and pH is analogous to that found for cellulose by Clibbens and Ridge; i.e. the rate is a maximum at a point between pH 6 and 8 and falls off sharply on each side. Ethanol behaves similarly above pH 4, but reacts vigorously at low pH owing to its great reactivity towards free chlorine. Glucose and glycollaldehyde behave quite differently; the reaction mechanism is indicated briefly. The kinetics of the reactions of lactic and D-gluconic acids for any given pH in the range 6–10 is represented by an empirical equation of the second order—

$$-\frac{dc}{dt} = kAC$$

where A is the concn. of the hydroxy acid ion, and C is the concn. of active chlorine, i.e. the sum of $[OCl^-]$ and [HOCl]. The kinetic behaviour over the range of pH cannot be represented by any equation in which $[OCl^-]$ and/or [HOCl] appear as factors to the first or higher power. The mechanism proposed is as follows— The hydroxy cpd. reacts with hypochlorous acid forming an unstable hypochlorous ester—

$$>CH\cdot OH + HOCl \rightarrow >CH\cdot OCl + H_2O \qquad (i)$$

This reacts with hydroxyl ions forming a ketone or aldehyde—

$$>CH\cdot OCl + OH^- \rightarrow >CO + H_2O + Cl^- \qquad (ii)$$

At a low pH, when [HOCl] is high and $[OH^-]$ is low, (ii) is the rate-determining reaction, and the rate rises approx. in proportion to $[OH^-]$. On passing over to the alkaline side, (ii) becomes more rapid and ceases to limit the overall rate, which becomes more and more dependent on the rate of (i), which is determined by [HOCl] and therefore falls with a further rise in pH. The empirical second-order equation is obeyed at a given pH because C is proportional to [HOCl]. The oxidation of cellulose by hypochlorite solutions is considered to proceed by the same mechanism. On the other hand, the oxidation of dyes by such a soln. generally follows the pattern of aromatic substitution or addition at double bonds; the active agents are Cl_2, Cl_2O, and HOCl, Cl_2 reacting much more vigorously than HOCl. In this case, too, there may be a maximum near the neutral point, owing to the appearance of a more active form of dye in alkaline soln., but when the dye is adsorbed on the fibre, the maximum often disappears. The curve for the time required to bleach dyed cotton is superimposed on a curve showing the degree of oxidation of the cellulose resulting from the bleach, the common abscissa being the pH. It is readily seen from this diagram that for this particular case optimum conditions for bleaching with minimum damage to the fibre exist in the pH ranges 2–4 and 8·5–10, while the range 6–8 constitutes a danger zone where the same degree of bleaching is accompanied by considerable fibre degradation. The advantages of using the acid bleaching range are stressed, and it is recommended that modified processing plant should be constructed to enable chlorine-water bleaching to be carried out on the large scale. A. E. S.

Hydrogen Peroxide Dry-in Process for Bleaching Wool. E. S. Shanley, H. O. Kauffmann, and W. H. Kibbel. *Amer. Dyestuff Rep.*, **40**, 1–4 (8th Jan. 1951).

Wool is impregnated in a scouring bowl at the end of the usual scouring train with a solution of 1–3-vol. H_2O_2, acidity being adjusted so that the wool is neutral or faintly alkaline after subsequent drying. A typical 40% reflection of blue light by a medium coloured wool can thus be increased to 45–50%, light blue and light grey being particularly brighter. Residual H_2O_2 has, however, a serious effect, and can be removed by 0·2% (on wt. of wool) sodium hydrosulphite, by a yeast slurry to catalyse decomposition, or by adding small amounts of reducing agent to the dyebath. Production of clear shades is particularly assisted by dyeing at 80°C. J. W. B.

PATENTS

Chlorite and Hypochlorite Bleaching of Cellulosic Textiles. Mathieson Chemical Corpn. and R. L. Carr. *U.S.P.* 2,521,340.

Impregnate with an aqueous solution of sodium chlorite and hypochlorite and then steam in absence of excess solution. Under such conditions the hypochlorite is not rapidly decomposed in presence of the chlorite, evolution of chlorine dioxide is inhibited, and the goods are rapidly bleached without deleterious effects. C. O. C.

Cloth Bleaching. Minneapolis-Honeywell Regulator Co. and H. M. Schmitt. *U.S.P.* 2,522,900.

In the continuous bleaching of cloth the volume is regulated while the composition of the liquor is maintained constant by varying the rates at which (*a*) water is brought into the bath by the cloth and (*b*) bleaching agent is passed into the bath. The variations in these rates are controlled by variations in the level of the liquor. The cloth is squeezed as it leaves the bath, so that the rate at which it takes liquor away from the bath is constant. C. O. C.

Bleaching Bast Fibres. I.C.I. Ltd., W. A. S. White, and N. F. Crowder. *B.P.* 648,632.

In chlorite bleaching of linen, etc., a good and permanent white can be obtained together with reduced chlorite consumption by first scouring with hot alkali, chemicking with acidified hypochlorite solution, then immersing in a mildly alkaline solution preferably containing an antichlor, and finally treating with a hot solution of an alkali or alkaline-earth metal chlorite at pH 6. The material is rinsed between each of these processes. C. O. C.

Stripping Wool or Rendering it more Resistant to Reducing, Oxidising, and Alkaline Agents. Harris Research Laboratories, M. Harris, C. W. Harris, L. E. Fourt, and A. M. Sookne. (X, p. 168.)

VIII— DYEING

Review of Textile Colouring and Finishing Research. G. L. Royer. *Amer. Dyestuff Rep.*, **39**, P 877–P 887 (11th Dec. 1950).

A review is presented of recent scientific contributions on the dyeing of wool, acetate, nylon, cotton, and viscose rayon and on the printing and finishing of textiles. Stress is laid on the need for more liaison between pure and applied research and between these and the application of practical methods. There are 145 references. J. W. B.

Weber–Fechner Law and Dyeing Strengths. I. H. Godlove. *Amer. Dyestuff Rep.*, **40**, P 49–P 50 (22nd Jan. 1951).

In a criticism of a paper on the application of psychophysical principles to textile dyeing (H. Cikurel, *ibid.*, **39**, P 736 (30th Oct. 1950)), it is considered that there is not a strict relation between dye strength on a fibre and visual stimulus and that, therefore, the Weber–Fechner law cannot be accurately employed to correlate the amount of dye on a fibre and equal visual steps. J. W. B.

Comparison of the Dyeing Characteristics and Related Properties of Rain-grown and Irrigated Cotton. A.A.T.C.C., Piedmont Section. *Amer. Dyestuff Rep.*, **39**, P 901–P 911 (25th Dec. 1950).

Comparative analyses are made of Deltapine (rain-grown) and Acala (irrigated) cottons with regard to metallic constituents, pectic substances, ash and ash alkalinity, cold- and hot-water-extractable substances, degree of polymerisation, and rate of attack by HCl and bleaching agents. No difference is found except in the amount of water-extractable matter and in chain length, and experiments show that these factors are not related to the final depth obtained with various dyes. Despite quite material differences in depth being evident, spectrophotometric analyses show that the amount of dye absorbed by the two cottons is identical, while cross-section photomicrographs show similar distribution of dye. Differences are, however, accentuated or diminished by altering the incidence of illumination and also by mercerising the yarns, and it is therefore concluded that

reflectance from dyed fibre surfaces is responsible for the apparently different dyeing properties of these cottons. J. W. B.

Relations between the Constitution and Suitability for Metachrome Dyeing of *oo'*-Dihydroxyazo Dyes. G. Schetty. *Textil-Rund.*, **5**, 486 (Nov. 1950). Correction to previous paper, *Textil-Rund.*, **5**, 399–405 (Oct. 1950): J.S.D.C., **67**, 42–43 (Jan. 1951).

Increasing the Fastness of Dyeings on Wool Fabrics. M. Mayants. *Tekstil. Prom.*, **10**, 28–29 (Aug. 1950). OP–10, which is a condensation product of alkylphenols with ethylene oxide and is normally used as a wetting agent and detergent, is recommended for application to dyeings of acid, chrome, and direct dyes on wool and union fabrics as a means of increasing rubbing fastness. A. E. S.

Cheese Dyeing. P. Guillermin. *Teintex*, **15**, 555–562 (Dec. 1950). A practical paper giving the points to be watched and pitfalls to be avoided during the various stages in cheese dyeing. The use of rapid drying is compared with hydro-extraction followed by slow drying at each stage. Apart from winding and loading problems, attention is paid to bleaching and dyeing with special reference to oxidation, hydroextraction, and drying. B. K.

Continuous Dyeing of Nylon Fabrics. A.A.T.C.C., Rhode Island Section. *Amer. Dyestuff Rep.*, **40**, P 14–P 29, P 32 (8th Jan. 1951). Production of fast-dyed nylon piece, free from warpy or barry effects, has been made possible by laboratory and plant investigations. Dyeing takes place in two stages—(1) adsorption of dye without chemical bonding, and (2) diffusion into the fibre, allowing chemical bonding to take place. This theory is applied in practice by a padding process, which is followed by steaming to produce bonding. Complete fixation is not attained, but as no migration is observed during drying further treatment can be employed. Each dye reacts differently to the effect of assistants, steaming time, and dye concentration; hence individual studies are necessary for each dye. Some blocking effects in compound shade dyeing, i.e. preferential adsorption of one dye, are eliminated. Details of steaming time, shade build-up, effect of heat setting, and fastness properties are tabulated for several experiments using numerous dyes. J. W. B.

Dyeing of New Synthetic Fibres. P. L. Meunier. *Amer. Dyestuff Rep.*, **40**, P 51–P 54 (22nd Jan. 1951). Dyeing properties of continuous and staple Orlon acrylic fibre and of Fibre V from terephthalic acid and ethylene glycol are discussed. Under ordinary conditions Orlon has a moderate affinity only for acetate rayon, vat, and basic dyes, and may therefore be used to produce undyed effect threads. Improved application of vat acids, soluble vat, and thioindigoid vat dyes at 212° and 250°F. is described. Orlon staple has much greater dye affinity than filament, and may be dyed also with acid, chrome, and direct dyes, the first two having light and wet fastness equal to those of the same dyes on wool. Fibre V in both forms may be dyed at 212°F. with dispersed acetate dyes in the presence of carriers, e.g. benzoic acid, while at higher temp. vat dyes and some naphthanil combinations are adsorbed. Improved light fastness is obtained with mixed acetate and vat dyes applied by the Thermosol process, which involves padding, drying, a short dry-heat treatment at 400°F., and soaping. J. W. B.

PATENTS

Dyeing of Textiles with Dyed Cellulose Ethers. Dan River Mills Inc. *B.P.* 649,140. Uniform dyeing is obtained by first applying the dye to a cellulose ether, which is then dissolved in alkali. This solution is then applied to the material to be dyed, which is finally treated with acid to precipitate the dyed cellulose ether on to it. C. O. C.

Wool Dyeings metallised with Aluminium, Manganese, Iron, Cobalt, Nickel, Copper, Tin, or Zirconium. American Cyanamid Co., G. L. Royer, and C. A. Amick. *U.S.P.* 2,520,106. Wool, silk, and other fibres containing basic nitrogen are dyed very level and in brilliant hues from baths containing a metallisable dye and a soluble salt of one or more of the above metals. The hues obtained are entirely different from those produced by the use of chromium salts. The hydrolysis of the tin or zirconium salts does not interfere with the dyeing, particularly if a cationic or non-ionic surface-active agent is present. Preferably the bath should be alkaline at the start (pH not $> 9{\cdot}5$), the pH being gradually lowered as dyeing proceeds. C. O. C.

Increased Strength and Levelness in Metachrome Dyeing. American Cyanamid Co., H. E. Millson, C. A. Amick, and W. H. Watkins. *U.S.P.* 2,520,105. Markedly increased strength and levelness of dyeing are obtained if there is present in the dyebath an alkaline-earth metal salt and a cationic surface-active agent and if the initial pH is sufficiently high to prevent or retard chromation, the pH being gradually lowered during dyeing until final chromation occurs. C. O. C.

Dyeing Protein Fibres with Chromable Anthraquinone Dyes by the Metachrome Process. American Cyanamid Co., W. H. Watkins, and C. A. Amick. *U.S.P.* 2,520,108. Modification of *U.S.P.* 2,520,105 (above). With chromable anthraquinone dyes marked increase in colour value is obtained if the alkaline-earth metal salt and initial high pH followed by reduced pH are used in absence of cationic surface-active agents. C. O. C.

Metachrome Dyeing with Azo Dyes containing Nitrogen-free Metallisable Groups *ortho* to the Azo Group. American Cyanamid Co., C. A. Amick, W. H. Watkins, and H. E. Millson. *U.S.P.* 2,520,080. When using azo dyes which have no nitrogen-containing group *ortho* to the azo group, e.g. *C.I.* 203, marked increase in colour value is obtained if an alkaline-earth metal salt, e.g. $MgSO_4$ or $Ca(NO_3)_2$, and initial high pH followed by a reduced pH are used in absence of cationic surface-active agents. In addition there is no scum or floc formation. C. O. C.

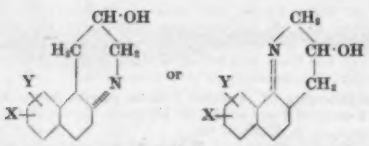
Increased Colour Value with Metachrome Dyeings. American Cyanamid Co., C. A. Amick, and G. L. Royer. *U.S.P.* 2,520,081. Improved colour value, levelness, penetration, and exhaustion are obtained in metachrome dyeing if there is present in the dyebath a colloidal cationic surface-active agent, a soluble compound of a metal of Group IIB, e.g. Zn or Cd, and if the dyebath is alkaline (pH not $> 9{\cdot}5$) at the start and is gradually acidified until it has pH 4·0 at the end of dyeing. A number of dyes not suitable for the ordinary metachrome process are commercially applicable by this method. C. O. C.

Colouring Cellulose Esters and Ethers with Vat Dyes. H. C. Olpin and E. Stanley. *B.P.* 647,897. An aqueous bath containing the leuco vat dye and an organic base which has a substantial swelling action on the cellulose ester or ether, e.g. 15–25% of pyridine on the total weight of the liquor, is used. By this means vat dyes containing two or more anthracene nuclei, e.g. Caledon Jade Green B, Red X5B, Brown G, and Brilliant Blue 3G (ICI), can be satisfactorily applied to acetate rayon without material saponification. *B.P.* 647,898. The same advantages are obtained if the bath is acid, neutral, or faintly alkaline and the organic base is replaced by a water-miscible organic liquid which is a strong swelling agent for acetate rayon. C. O. C.

Colouring Organic Derivatives of Cellulose. British Celanese Ltd. *B.P.* 648,725. Very rapid dyeing with a wide range of dyes is obtained by mechanical impregnation with an aqueous dye liquor containing a lower aliphatic acid and an inorganic swelling agent. The material may be rinsed within a very short time of the impregnation. C. O. C.

Inhibiting Gas Fading of Coloured Organic Derivatives of Cellulose. General Aniline & Film Corpn., H. W. Grimmel, and J. F. Morgan. *B.P.* 648,631. *N*-Substituted derivatives of 5-aminotetrazole and of 3-amino-1:2:4-triazole, e.g. "α-dibenzyl-5-aminotetrazole" (probably 2-benzyl-5-benzylaminotetrazole) or dibenzyl-3-amino-1:2:4-triazole, are excellent gas-fading inhibitors applicable in the dyebath. C. O. C.

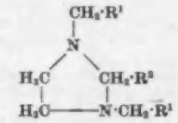
Naphthopyridine Derivatives as Gas Fading Inhibitors. General Aniline & Film Corpn., H. W. Grimmel, and A. Guenther. *B.P.* 646,832. Compounds of formula—



(X and Y = H, Alk, Hal, cyano, or nitro) are good gas fading inhibitors for use on cellulose esters, being particularly suitable for application in the dyebath. C. O. C.

Iminazolidine Derivatives as Gas Fading Inhibitors. General Aniline & Film Corpn., H. W. Grimmel, and A. Guenther. *B.P.* 648,651.

Compounds of formula—



(both R^1's are the same and R^1 and R^2 = aliphatic, halogenoalkyl, *cyclo*alkyl, aralkyl, aromatic, halogenoaryl, alkoxyaryl, alkylaryl, or heterocyclic radicals), e.g. 2-phenyl-1:3-dibenzyliminazolidine, are excellent gas-fading inhibitors, particularly suitable for application during dyeing. C. O. C.

Dyeing Cellulose–Cellulose Ester Mixtures with Vat Dyes. Celanese Corpn. of America, G. C. Ward, and W. T. Haggerty, Jr. *U.S.P.* 2,524,092.

Rapid and continuous dyeing is satisfactorily achieved by treating the material with an aqueous vat, at pH 10·5–11·5 and not < 40°c., which contains a thiocyanate and 20–50% by weight of diacetone alcohol. The material is rinsed directly after immersion and then oxidised before drying, again rinsed, soaped, and dyed. Solid dyeings are obtained with 20–35% of diacetone alcohol; above that concentration the cellulose ester is dyed but not the cellulose. C. O. C.

Dyeing Cellulose Ester–Animal Fibre Mixtures with Vat Dyes. Celanese Corpn. of America, G. C. Ward, and W. T. Haggerty, Jr. *U.S.P.* 2,524,093.

Solid dyeings are rapidly and continuously produced by passing the material through the vat, which is kept at pH 10·5–11·5 and which contains a thiocyanate and 10–30% by weight of diacetone alcohol, the temperature of the vat being 55–65°c. The material is rinsed immediately after immersion, oxidised without drying, rinsed, soaped, and dried. C. O. C.

Dyeing or Printing Nylon. Ciba Ltd. *B.P.* 646,742.

Acid and direct cotton or other water-soluble dyes containing acid groups are applied to nylon together with a salt of a volatile base and a less volatile acid and/or with a heat-decomposable ester of an acid, e.g. ammonium tartrate, and the dyed or printed material is then steamed. C. O. C.

Deep Black Surface Coloration of Cadmium. United Chromium Inc. and J. E. Stareck. *U.S.P.* 2,524,577.

A deep black coloration having good adhesion is obtained on cadmium by treatment in a solution containing a sexivalent chromium compound, a silver salt, and sulphuric or hydrofluoric acid or a sulphate or a fluoride. C. O. C.

Cultivation of Coloured Cotton. A. Lübke. (VI, p. 162.)

Emulsions for use in Fabric-decorating. Interchemical Corpn. and A. Booth. (IX, this page.)

IX—PRINTING

Printing Cotton Fabrics with Direct Dyes. V. Rostovtsev. *Tekstil. Prom.*, 10, 26–28 (Aug. 1950).

Fast, bright prints of direct dyes may be obtained by making an addition to the printing paste of a soln. obtained by adding caustic soda to copper sulphate soln. in presence of glycerol. After steaming, the print is passed through a hot soln. of Fixer DCU (dicyandiamide–formaldehyde condensate). Alternatively, the dye may be printed with dicyandiamide, ammonium chloride, triethanolamine, and formaldehyde. In this case the fabric, after steaming, is passed through a hot soln. of Fixer DCM (copper deriv. of DCU). The second method yields the brighter results. A. E. S.

PATENTS

Printing with Sulphuric Esters of Leuco Anthraquinone Vat Dyes. General Aniline & Film Corpn. and A. Schoen. *U.S.P.* 2,521,485.

Complete inhibition of development by steaming for 1–3 min. and then inducing an autocatalytic or chain reaction generating an acid state renders possible the production of prints, even on rayon, of high density and excellent resistance to washing. Development may be inhibited by replacing the usual acid splitters by an organic sulphur compound incapable of splitting a salt or ester under the conditions which prevail during development, e.g. an aldehyde-sulphite or -sulphoxylate. C. O. C.

Printing, Cover Pad Printing, and Padding. L. Krause and J. A. Wainwright. *B.P.* 647,098.

Dyes and/or discharges, which can be fixed on the material by ageing or steaming, can be applied to fabrics in printing or padding pastes which rely not upon thickeners or similar film-forming substances for their consistency but upon liquid–liquid emulsions of the oil-in-water or of the water-in-oil types. Use of such pastes enables screens and rollers to be cleansed much more easily than when conventional printing pastes are used. Furthermore, where dyes and/or discharges are used which are substantive to the fibre under ordinary dyeing conditions, no aftertreatment is necessary after steaming or ageing. C. O. C.

Emulsions for use in Fabric-decorating. Interchemical Corpn. and A. Booth. *B.P.* 647,251.

An inner aqueous phase contains a dispersed water-insoluble film-forming resin and an outer, water-immiscible organic phase consists essentially of a solution of film-forming binder in a volatile organic solvent in which the resin in the other phase is insoluble. The use of such an emulsion for pigment-printing or dyeing fabrics gives products which are fast to rubbing without imparting an objectionable handle or boardiness. In addition, better colour value is obtained than with the hitherto known methods. C. O. C.

Lead Chromate Photographic Images. General Aniline & Film Corpn. and D. E. Sargent. *B.P.* 648,591.

The silver thiobarbiturate image obtained by treating a silver halide image with 2-thiobarbituric acid is readily converted into a lead chromate image by treatment first with an acid aqueous solution of a lead salt and then with an aqueous solution of a chromate. C. O. C.

Diazotype Photoprinting Materials. General Aniline & Film Corpn., W. H. von Glahn, and L. N. Stanley. *B.P.* 647,617.

Addition of colloidal silica to diazotype coating compositions prevents undue penetration into the paper and has no adverse effects. C. O. C.

Diazotype Photoprinting Materials. General Aniline & Film Corpn. and H. C. Unkauf. *B.P.* 647,620.

Use of coupling components of formula—

$$R^1{\cdot}NR^3{\cdot}CO{\cdot}CH_2{\cdot}COOR^2$$

(R^1 and R^2 = H, Alk, hydroxyalkyl, or aralkyl; R^3 = Alk, Ar, or aralkyl), e.g. methyl or ethyl malonamate, yields diazotype compositions stable to precoupling. C. O. C.

Printing by the Action of Heat on Heat-sensitive Materials. J. Groak. *B.P.* 647,821.

Mono- or hetero-chromatic prints are obtained by bringing into contact two surfaces, one of which consists of a composition which softens on heating, this surface being scanned before or during the contact between the two surfaces with a controlled radiant energy beam. This beam consists of either electrically charged corpuscles or radiant heat. This softens part of this surface, so that material is transferred from its softened parts to the other surface.

B.P. 647,831.

The composition which softens on heating can change its nature permanently when heated, e.g. it may become coloured or change its colour. C. O. C.

Ceramic Colour Vehicle Compositions for use in Screen Printing. B. F. Drakenfeld & Co. Inc. and R. Andrews. *B.P.* 648,209.

A vitrifiable glaze is dispersed in a heat-liquefiable thermoplastic resin. The composition is normally solid, but liquefies readily on heating, so that it can be applied by screen printing. As it solidifies quickly upon contact with the surface to be printed, another colour can be applied without intermediate drying or heating. C. O. C.

Magenta Phenazonium Dye Images. General Aniline & Film Corpn., V. Tulagin, and R. F. Coles. (IV, p. 161.)

Review of Textile Colouring and Finishing Research. G. L. Royer. (VIII, p. 165.)

Dyeing or Printing Nylon. Ciba Ltd. (VIII, p. 167.)

X—SIZING AND FINISHING

Conditioning of Rayon Staple by High-frequency Heating. H. Vettermann. *Textil-Praxis*, **6**, 18–19 (Jan. 1951).

Accurate control of the moisture content (condition) of rayon staple is important for subsequent spinning. The standard method at present employed in Germany necessitates 2·5 hr. drying for a 200–250 g. sample. An alternative method is described which employs high-frequency drying and claims to give accurate results after 6–7 min. drying. B. K.

Application of Vinyl Polymers to Textiles. F. Weiss. *Textil-Rund.*, **5**, 487–497 (Dec. 1950).

A review of the properties of vinyl polymers and of their application to textiles in the form of aqueous dispersions. F. A.

Prediction of the Bactericidal Effects of Substances by Determination of their Affinities for Wool. R. Fischer, A. Hoelle, and S. Seidenberg. *Helv. Chim. Acta*, **34**, 210–222 (Feb. 1951).

The conclusions drawn by subjecting 18 substances of anionic, cationic, and non-ionic character to the procedure already detailed (*ibid.*, **32**, 8 (1949) and *Experientia*, **6**, 192 (1950)) are as follows—(1) Increasing affinity for wool (from neutral aqueous solution at 90°C. for 10 min.) is accompanied by a similar rise of effect on Gram-positive bacteria. (2) By systematic degradation of wool at 80°c. with 0·15 N-Na_2CO_3, wool protein stages can be obtained whose pH values correspond with the isoelectric points of Gram-positive and Gram-negative bacteria respectively. (3) It is possible in principle to forecast Gram-negative bactericidal power by comparison of the affinities for wool of the substances at these two stages of degradation. (4) Thus the bactericidal power of any water-soluble thermostable substance can be predicted. (5) Non-ionic substances, which generally exhibit no bactericidal effect, may, however, become bacteriostatically active. H. H. H.

PATENTS

Rendering Wool Shrink-resistant. Eavenson & Levering Co., American Cyanamid Co., E. N. Angus, J. W. Creely, and R. M. Fischer. *U.S.P.* 2,522,338.

The reduction of the felting power of wool imparted by treatment with a thermosetting resin is seriously impaired by extensive mechanical working, which causes fibre displacement. This is overcome by impregnating the wool with the precondensate of the resin, then giving it the necessary mechanical working, e.g. carding, knitting, or weaving, and subsequently curing the resin. C. O. C.

Stripping Wool or Rendering it more Resistant to Reducing, Oxidising, and Alkaline Agents. Harris Research Laboratories, M. Harris, C. W. Harris, L. E. Fourt, and A. M. Sookne. *B.P.* 646,809.

The wool is treated with a reducing agent for keratin, e.g. sodium hydrosulphite or foramidinesulphinic acid, in presence of free formaldehyde where modification of the keratin alone is desired, or in presence of an alkylene dihalide, e.g. 1:2-dibromoethane, where stripping is required as well as modification of the keratin. The physical properties of the fibre are unaffected by the treatment. C. O. C.

Stiffening of Wool Fabrics. Trubenised Ltd. and E. Higgins. *B.P.* 646,930.

A woven or knitted interlining or backing comprises wool yarn and thermoplastic yarn, e.g. acetate rayon, both sets of yarns having the same diameter. The fabric structure and the number and position of the thermoplastic yarns are so chosen that, after the interlining or backing has been bonded to a wool fabric by heat and pressure, the assembly is still permeable. C. O. C.

Straightening of Animal Fibres particularly in the Form of Furs and Pile Fabrics. Lister & Co. Ltd. and G. B. E. Schueler. *B.P.* 648,854.

A thermohardening or thermosetting synthetic resin or its precondensate or components are applied to the material, and then the fibres or pile vigorously brushed while heating at 100–160°c., so as to straighten the fibres while simultaneously curing the resin. C. O. C.

Rendering Cotton–Alkali-insoluble Artificial Fibres Yarns Free from Fuzz and More Absorbent. Textile Machinery Corpn. *B.P.* 648,815.

Yarns of mixtures of cotton and alkali-insoluble artificial fibres, e.g. acetate rayon or nylon, are freed from fuzz and rendered more absorbent by treating them for 90–120 sec. in 22% aq. NaOH at 75–80°F., washing, neutralising, and drying. C. O. C.

Stabilising Cellulosic Textiles to Shrinkage employing Glyoxal and a Metal Silicofluoride Catalyst. Alrose Chemical Co. and L. Beer. *U.S.P.* 2,521,328.

Textiles formed predominantly of cellulosic fibres are stabilised to shrinkage by treating them with dilute aqueous solutions of glyoxal using a soluble inorganic silicofluoride as catalyst. There is no appreciable loss in tensile strength or abrasion resistance. C. O. C.

Flameproofing Cellulosic Materials. American Cyanamid Co., A. M. Loukomsky, R. H. Kienle, and T. F. Cooke. *U.S.P.* 2,519,388.

A flameproof, which is fast to washing and does not alter the handle of the material, is obtained by impregnation with a mixture of (*a*) an oxide of Sn, Ti, Sb, or Bi (1 part by weight) and a thermoplastic substance containing at least 20% of combined halogen and rupturing at a C–Hal bond when heated, e.g. polyvinylidene chloride (0·6–20·0); and (*b*) a water-sol. salt of a quinquevalent phosphorus oxyacid (1, calc. as H_3PO_4) and urea, biuret, ammonium cyanate, or ammonium dicyanamide (1–20), the proportion of (*a*) : (*b*) being 1 : 0·2–8·0.

U.S.P. 2,520,103.

American Cyanamid Co., M. R. Burnell, and W. J. van Loo, Jr.

The metallic oxide used is antimony trioxide.

U.S.P. 2,519,348.

A 65–85% aqueous dispersion of the composition described in *U.S.P.* 2,519,388 (*above*) is rendered stable to long storage and dilution with water by addition of 4% by weight of 28% aq. NH_3. C. O. C.

Use of Ultrasonic Vibrations in Washing Textiles. S. B. Wiczer. (I, p. 154.)

Chemically Modified Starches. United States Rubber Co., D. L. Schoene, and V. S. Chambers. (III, p. 155.)

Relationship between Structure and Creasing Properties of Viscose Rayon—I. E. Elöd and M. Kramer. (VI, p. 163.)

Review of Textile Colouring and Finishing Research. G. L. Royer. (VIII, p. 165.)

XIII—RUBBER; RESINS; PLASTICS

Emulsions, Dispersions, and Solutions of Methacrylic Ester Copolymers. Vinyl Products Ltd., J. E. O. Mayne, and H. Warson. (III, p. 157.)

Fibres, Films, etc. of *N*-Fluoroacrylamide Copolymers. Eastman Kodak Co., H. W. Coover, Jr., and J. B. Dickey. (VI, p. 163.)

XIV—ANALYSIS; TESTING; APPARATUS

Ionisation of Triarylcarbinol Indicators in Sulphuric Acid–Water Mixtures. M. A. Murray and G. Williams. (IV, p. 158.)

Weber–Fechner Law and Dyeing Strengths. I. H. Godlove. (VIII, p. 165.)

Prediction of the Bactericidal Effects of Substances by Determination of their Affinities for Wool. R. Fischer, A. Hoelle, and S. Seidenberg. (X, this page.)

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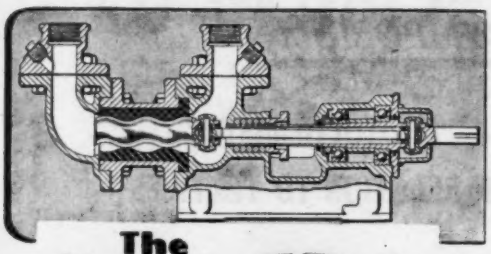

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Situations Vacant—*continued*

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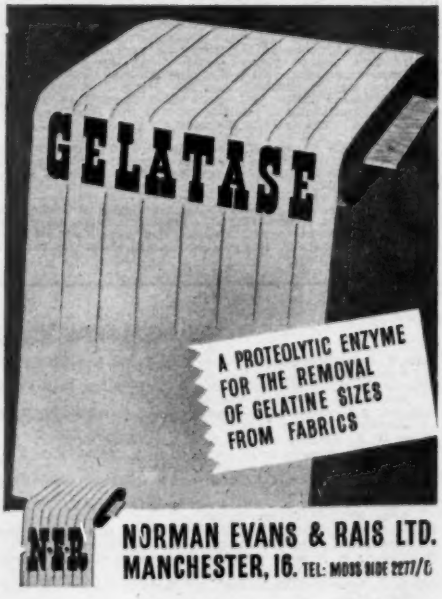

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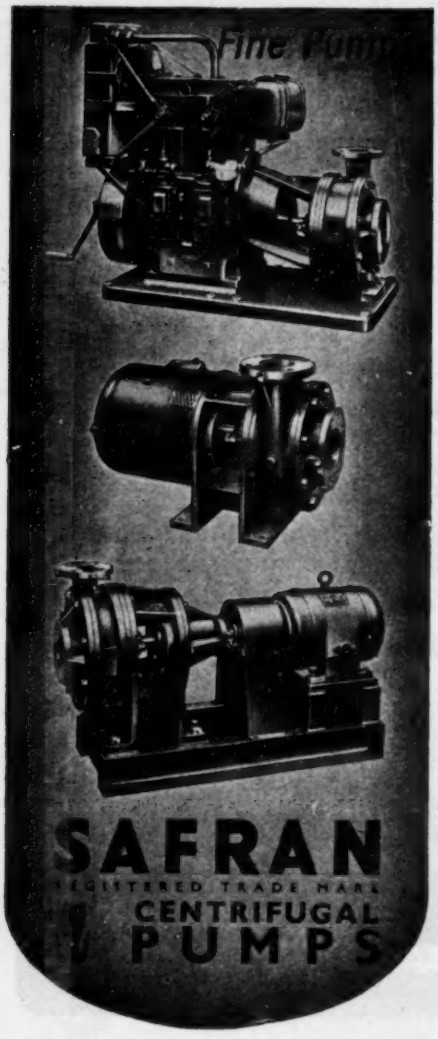

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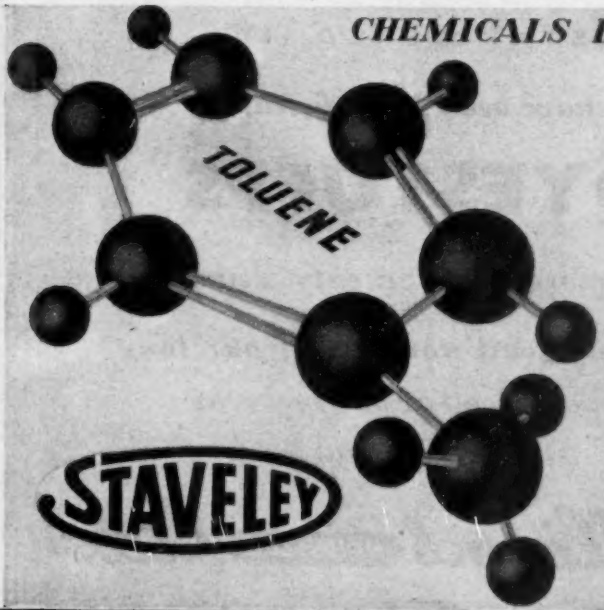

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